

**STANDARD METHODS FOR TESTING
TAR AND ITS PRODUCTS
1938**

STANDARDIZATION OF TAR PRODUCTS
TESTS COMMITTEE

Standard Methods for Testing Tar and its Products

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The Standardization of Tar Products Tests Committee was organized under the auspices of the Joint Fuel Committee, representing the following organizations:—

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SOCIETY OF CHEMICAL INDUSTRY

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PREFACE

THE present volume represents a thorough revision of the 1929 edition.

The revision of the several main sections of the book was allocated to Groups under the Revisionary Panel as indicated on page viii. The Revisionary Panel was responsible for the general co-ordination. The entire Committee has been fully consulted on the revised text, which is issued on its authority.

The Committee wishes to record its high appreciation of the work of the Revisionary Panel and Panel Groups in the preparation of the revised text and in the laboratory investigations on which the revision is based. The Committee's thanks are also due to the firms which have borne the cost of the laboratory work and of the attendance of members at meetings.

The Committee is indebted to the American Wood Preservers' Association and the Treasury Department of the United States Government for permission to reproduce certain of their publications; to the Institute of Petroleum for permission to use the contents of "Standard Methods for Testing Petroleum and its Products" as far as the Committee desired, with the object of securing correlation between the two industries; and to the National Physical Laboratory for invaluable help in the preparation of the sections on specific gravity and density and of the apparatus schedules.

So far as possible, the scheduled apparatus units are those specified by the British Standards Institution; these in turn are in the majority of cases based on the Committee's 1929 standard apparatus.

It is confidently believed that this edition will prove more valuable than was the 1929 edition to the producers of coal tar and its products, the consumers and their associates alike. The Committee will welcome comments from users of the book, whose views will be valuable in the future work of the Committee.

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CONTENTS

	SERIAL NO.	PAGE
Preface		v
1. GENERAL PRINCIPLES		
Introduction		1
Definitions		7
Sampling		13
Density and Specific Gravity		31
Density	G.P. 1-38	31
Specific Gravity	G.P. 2-38	47
Distillation Principles	G.P. 3-38	62
2. CRUDE TAR		
ANALYSIS FOR COMMERCIAL EVALUATION		
Nature and Quantity of Sample		71
Preliminary Treatment of the Sample		71
Specific Gravity and Density of the Tar as received	C.T. 1-38	72
Specific Gravity and Density of the Anhydrous Tar	C.T. 2-38	72
Water	C.T. 3-38	73
Insoluble Matter ("Free Carbon")	C.T. 4-38	74
Chlorine (calculated as Ammonium Chloride)	C.T. 5-38	75
Ash	C.T. 6-38	76
Distillation (Full Scale)	C.T. 7-38	77
Distillation (Small Scale)	C.T. 8-38	92
3. REFINED TAR, INCLUDING ROAD TAR		
Nature and Quantity of Sample		97
Preliminary Treatment of the Sample		97
Specific Gravity; Density	R.T. 1-38	97
Viscosity	R.T. 2-38	97
Equi-viscous Temperature (E.V.T.)	R.T. 3-38	104
Water	R.T. 4-38	109
Distillation	R.T. 5-38	110
Phenols	R.T. 6-38	113
Naphthalene	R.T. 7-38	115
Insoluble Matter ("Free Carbon") and Ash	R.T. 8-38	115
4. CRUDE BENZOLE		
Applicability		117
Nature and Quantity of Sample		117
Preliminary Treatment of the Sample		117
Preliminary Tests—		
Specific Gravity; Density	C.B. 1-38	117
Distillation	C.B. 2-38	118
Total Sulphur	C.B. 3-38	120
Carbon Disulphide	C.B. 4-38	121

CONTENTS

	SERIAL NO.	PAGE
Commercial Analysis—		
Loss on Acid-washing	C.B. 5-38	121
Fractionation	C.B. 6-38	123
Determination of Pure Products .. .	C.B. 6a-38	123
Determination of Commercial Fractions	C.B. 6b-38	124
 5. LIGHT AND MIDDLE OILS		
Applicability		127
Nature and Quantity of Sample		127
Preliminary Treatment of the Sample		127
Preliminary Tests—		
Specific Gravity; Density	L.M. 1-38	128
Water	L.M. 2-38	128
Distillation	L.M. 3-38	129
Phenols	L.M. 4-38	130
Commercial Analysis	L.M. 5-38	132
Phenols (Removal and Recovery)	L.M. 5a-38	133
Pyridine Bases	L.M. 5b-38	135
Crude Naphthalene	L.M. 5c-38	137
Loss on Acid-washing; Refined Naphthas and Neutral Oils	L.M. 5d-38	138
 6. REFINED LOWER BOILING PRODUCTS		
Applicability		141
Nature and Quantity of Sample		141
(a) GENERAL		
Specific Gravity; Density	L.B. 1-38	141
Colour	L.B. 2-38	141
Flash-point	L.B. 3-38	142
Crystallizing-point	L.B. 4-38	145
Neutrality	L.B. 5-38	147
Distillation	L.B. 6-38	148
Phenols	L.B. 7-38	151
Pyridine Bases	L.B. 8-38	152
Acid Wash Test	L.B. 9-38	153
Total Potential and Existent Gum (Oxidation Test for Stability of Benzole during Storage)	L.B. 10-38	155
Existent Gum (Residue on Evaporation)	L.B. 11-38	160
Unsaturated Hydrocarbons (Bromine Absorption)	L.B. 12-38	160
Non-sulphonable Hydrocarbons (Paraffins and Naphthenes)	L.B. 13-38	162
Total Sulphur	L.B. 14-38	164
Carbon Disulphide (Qualitative Test)	L.B. 15-38	171
Carbon Disulphide (Quantitative Test)	L.B. 16-38	171
Hydrogen Sulphide and Mercaptans (Qualitative Test)	L.B. 17-38	174
Mercaptans (Quantitative Test)	L.B. 18-38	176
Corrosive Sulphur (Copper Strip Test)	L.B. 19-38	177
Corrosive Sulphur (Gravimetric Test)	L.B. 20-38	178
Thiophen (Qualitative Test)	L.B. 21-38	180
Thiophen (Quantitative Test)	L.B. 22-38	181

CONTENTS

xi

	SERIAL No.	PAGE
(b) SPECIAL FRACTIONS		
Benzene, Toluene and Xylene in Certain Mixtures	L.B. 23-38	183
Benzene and Toluene in Certain Mixtures ..	L.B. 23a-38	183
Benzene, Toluene and Xylene in Certain Mixtures	L.B. 23b-38	184
Xylene in Certain Mixtures	L.B. 24-38	186
Benzene, Toluene and Xylene in Commercial Toluole	L.B. 25-38	188
Metaxylene in Xylole	L.B. 26-38	191
Colman and Yeoman Test	L.B. 27-38	192

7. CARBOLIC AND CRESYLIC ACIDS

Nature and Quantity of Sample	203
---------------------------------------	-----

(a) GENERAL

Specific Gravity; Density	C.C. 1-38	203
Distillation	C.C. 2-38	203
Commercial Examination (Water Content, Modified Lowe's Test, and Residue on Distillation) of Crude Carbolic Acid	C.C. 3-38	205
Commercial Examination (Water Content, Phenol Content, and Residue on Distillation) of Crude Cresylic Acid	C.C. 4-38	208
Neutral Oils and Pyridine Bases	C.C. 5-38	212
Acidity and Alkalinity	C.C. 6-38	215

(b) PHENOLS AND REFINED CRESYLIC ACIDS

Water	C.C. 7-38	216
Crystallizing-point	C.C. 8-38	217
Residue on Evaporation	C.C. 9-38	218
Hydrogen Sulphide (Qualitative Test)	C.C. 10-38	219
Solubility of Phenol in Water	C.C. 11-38	220
Phenol in Liquefied Phenol	C.C. 12-38	220
Colour of Cresylic Acid	C.C. 13-38	222
Phenol in Phenol-Cresol Mixtures	C.C. 14-38	223
Phenol in Cresylic Acid	C.C. 15-38	226
Orthocresol in Cresylic Acid	C.C. 16-38	230
Metacresol in Cresylic Acid	C.C. 17-38	234

(c) HIGH BOILING PHENOLS

Water	C.C. 18-38	236
Phenols	C.C. 19-38	237
Cresylic Acid—Distillation (United States Customs Method)	C.C. 20-38	238

NAPHTHALENE

Nature and Quantity of Sample	247
Preliminary Treatment of the Sample	247

	SERIAL NO.	PAGE
(a) CRUDE NAPHTHALENE		
Water	C.N. 1-38	247
Crystallizing-point	C.N. 2-38	248
Oil and/or Naphthalene	C.N. 3-38	250
Phenolic Bodies	C.N. 4-38	252
Matter Insoluble in Toluole	C.N. 5-38	254
(b) REFINED NAPHTHALENE		
Crystallizing-point	R.N. 1-38	255
Sulphuric Acid Test	R.N. 2-38	256
9. TAR BASES		
Nature and Quantity of Sample		257
(a) CRUDE TAR BASES		
Preliminary Treatment of the Sample		257
Specific Gravity; Density	T.B. 1-38	257
Water and Refined Bases	T.B. 2-38	257
Neutral Oils	T.B. 3-38	259
Ammonia	T.B. 4-38	261
Ash	T.B. 5-38	262
(b) REFINED PYRIDINE BASES		
General		263
Specific Gravity; Density	R.P. 1-38	263
Colour	R.P. 2-38	263
Water	R.P. 3-38	263
Distillation	R.P. 4-38	264
Congo Red Test	R.P. 5-38	265
Cadmium Chloride Test	R.P. 6-38	265
Nessler's Reagent Test	R.P. 7-38	266
Miscibility with Water	R.P. 8-38	266
(c) REFINED HEAVY BASES		
Specific Gravity; Density	H.B. 1-38	266
Distillation	H.B. 2-38	267
Solubility in Acid	H.B. 3-38	267
10. CREOSOTE OIL		
Applicability		269
Nature and Quantity of Sample		269
Preliminary Treatment of the Sample		269
Specific Gravity; Density	C.O. 1-38	269
Water	C.O. 2-38	269
Distillation	C.O. 3-38	270
Phenols	C.O. 4-38	272
Solid Hydrocarbons	C.O. 5-38	274
Matter Insoluble in Toluole	C.O. 6-38	275
Ash	C.O. 7-38	276

CONTENTS

xiii

	SERIAL NO.	PAGE
Coke Residue	C.O. 8-38	276
Fluidity	C.O. 9-38	280
Flash-point	C.O. 10-38	282
Viscosity	C.O. 11-38	283
Calorific Value	C.O. 12-38	285
Total Sulphur	C.O. 13-38	293

Creosote Oil—Distillation (A.W.P.A. Method) ..	C.O. 14-38	294
--	------------	-----

11. CRUDE ANTHRACENE

Nature and Quantity of Sample	299
Anthracene	C.A. 1-38	299
Solid Paraffins	C.A. 2-38	302

12. PITCH

Nature and Quantity of Sample	305
Specific Gravity; Density	P.T. 1-38	305
Softening or Melting of Pitch—General	305
Twisting Test	P.T. 2-38	306
Kraemer and Sarnow Test (S.T.P.T.C. Modification) ..	P.T. 3-38	311
Ring and Ball Test	P.T. 4-38	317
Half Inch Cube Test	P.T. 5-38	322
Volatile Matter	P.T. 6-38	326
Insoluble Matter ("Free Carbon")	P.T. 7-38	329
Ash	P.T. 8-38	330

APPENDIXES

Appendix I: Apparatus Schedules	331
Appendix II: Physical Data	375
Index	429

1. GENERAL PRINCIPLES

INTRODUCTION

In the following pages, definitions and recommended methods for sampling are followed by the standard procedure for the tests more frequently applied—density, specific gravity and distillation; then follow the standard tests for tar and its products in the generally accepted order of sequence and, finally, appendixes of apparatus under schedule numbers and of physical data.

Copies of the graphs, printed on special paper suitable for mounting, are obtainable either individually or as a complete series from the office of the Committee at a reasonable charge.

Dimensions: The abbreviations recommended by the Comité International des Poids et Mesures have been adopted for metric dimensions, other abbreviations and symbols being taken in general from a British Standard list at present in course of preparation for chemical symbols and abbreviations.

The metric system of measurement has been adopted almost exclusively for apparatus, but trade practice has necessitated reference to imperial measures in certain tests, notably for crude tar.

Thermometric Scales: The Centigrade scale has been used for temperature measurements wherever possible, the only exceptions being in the flash-point test, where statutory requirements cannot be avoided; the viscosity test as applied to creosote, where the practice in the petroleum oil industry cannot be ignored; and the commercial examination of crude carbolic acid, where the long tradition of the Lowe's test would make it extremely difficult to effect a change from the Fahrenheit to the Centigrade scale.

The standard thermometers are in general specified as graduated for 100 mm immersion, but in one case a total immersion thermometer is specified so that the relevant test may conform in that particular with the practice abroad (see Serial No. P.T. 4, page 317).

It is essential that thermometers graduated for the same immersion are used by operators in testing to the same end; particular attention can advantageously be given to this point when international trade is involved, as foreign practice is frequently based on total immersion thermometers, if indeed the immersion has been standardized at all.

Reference Temperature: The Committee would have preferred to have adopted 20°C as the standard reference temperature for such purposes as specific gravity and density determinations, for the measurement of samples for test and of distillates, etc. The advantages of uniformity with, for example, the graduation of volumetric apparatus at 20°C do not appear however to be always commensurate with the disadvantages which would be introduced. Consequently, to take the particular instances mentioned, specific gravities $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ continue to be prescribed in general, and the measurement of samples etc. at laboratory temperature is retained.

In these circumstances, the temperature 20°C is to be regarded as the optimum temperature of test, but the operator must consider carefully whether the advantage to be gained in any particular case is likely to be less than the disadvantages which might accrue, for example from the additional manipulation of specially volatile products.

Volumetric Glassware: The Committee was represented on the committee of the British Standards Institution which prepared B.S. 554—1934: Report on Standard Temperature of Volumetric Glassware, and supported the recommendation that the temperature 20°C should be adopted as the standard temperature for British volumetric glassware. This temperature has been used in the British Standard specifications for a number of volumetric apparatus units which the Committee has adopted as far as possible in its standard tests. Similarly, 20°C has been adopted as the standard for such volumetric glassware as is peculiar to the testing of tar and its products.

Only an insignificant error is introduced by using interchangeably in the Committee's standard tests, volumetric glassware graduated either at 15°C or at 20°C . Thus, for example, the difference in capacity of two 1,000 ml flasks, one standardized at 15°C and the other at 20°C , would only be 0.13 ml when both are at the same temperature, whatever that temperature may be. Consequently, the volumetric glassware standardized by the Committee in the 1929 edition of "Standard Methods" on the basis of 15°C as the standard temperature may continue to be used without introduction of measurable error where the only difference from the 1938 specification is the temperature of graduation.

As stated on page 341, the Committee accepts as standard,

only apparatus conforming to the requirements of the National Physical Laboratory for Class A apparatus. It is recognized that it is not within the Committee's powers to require strict adherence to this principle in the internal routine work of the industry.

Unit of Volume: The Committee continues to use the millilitre as the standard metric unit of volume (see page 376).

Ground Glass Joints: The use of ground glass joints is in general highly to be recommended, especially if the interchangeable ground glass joints specified in B.S. 572—1934 are used, but the Committee considers that it would be unreasonable if they were made obligatory at this stage. Standard ground glass joints have therefore been prescribed only in those tests where their use is considered essential.

Density and Specific Gravity: Attention is drawn to the remarks on page 31 in regard to the Committee's position on the density versus specific gravity question.

Crystallizing-point/Melting-point: The temperatures determined by the standard tests are correctly described as crystallizing-points. In commercial practice the term "melting-point" is sometimes used incorrectly—this term should be reserved for the melting-point as determined by the normal capillary tube method or by the determination of the melting-point of a crystal floating on mercury. In general the crystallizing-point is lower than the melting-point, the difference being dependent on the purity of the material, the latent heat of fusion, etc.

Chemical Reagents: Chemical reagents are understood throughout to be of Analytical Reagent quality; the particular purpose for which a test is being carried out will determine the necessity or otherwise for close adherence to this ruling.

Percentages: The expression "*per cent*" is used according to circumstances with one of three different meanings. In order that the meaning to be attached to the expression in each instance may be clear, the following notation has been used where necessary:—

Per cent w/w (percentage, weight in weight) expresses the number of grams of active substance in 100 grams of product.

Per cent w/v (percentage, weight in volume) expresses the number of grams of active substance in 100 millilitres of product at 20°C.

Per cent v/v (percentage, volume in volume) expresses the number of millilitres of active substance in 100 millilitres of product at 20°C.

Limits of Accuracy: In cases where the expression of the limits of accuracy in percentages might lead to ambiguity, the Committee has adopted the practice of stating these limits in units; thus, if a "correct" result be X millilitres, and the limits of accuracy be given as $\pm Y$ units, the actual figure should fall within the limits $X - Y$ ml and $X + Y$ ml.

Where the limits of accuracy are given, the Committee is not able to state the allowance, if any, which shall be made in the analyst's report, this being a matter to be determined by specification, which is outside the terms of reference of the Committee.

Patented and Registered Apparatus: The Committee has, so far as possible, avoided the adoption of such apparatus as is known to be subject to patent or registration limitations. On the other hand, the Committee accepts no responsibility in the matter of limitations under this heading.

Standard Tests Omitted: The following standard tests, appearing in the 1929 edition of "Standard Methods" with the serial numbers and at the pages indicated in parentheses, have been omitted from the present edition and have not been replaced by other tests:—

Crude Tar—Treatment with Solvents (C.T. 5c, p. 45)

Insoluble Matter in Light Oil and Crude Benzole (L.O. 3, p. 101)

Analysis of Benzole Fore-runings (L.O. 5, p. 103)

Analysis of Pure Benzole (L.O. 8, p. 115)

Analysis of Toluole (L.O. 9, p. 117)

Toluene, Benzene and Xylene in Toluole (L.O. 10, p. 118)

Thiotolen in Toluole (L.O. 11, p. 122).

Inquiry has brought to light no evidence that the above-mentioned tests have been used to any significant extent in recent years and the Committee is of the opinion that the current edition of "Standard Methods" should be confined to tests which are in general demand. Nevertheless, the tests listed above are to be regarded as standard in the sense that when determinations based on the principles therein applied are required, the detailed standard method of test is that laid down in the 1929 edition; copies of the 1929 edition will be

kept available for sale by the Committee, price 7s. 6d. net, 8s. post paid.

The test for naphthalene in solvent naphtha, which appeared in the 1929 edition as Serial No. L.O. 13, page 125, has also been omitted, and is not replaced in the present edition. Neither that nor any other test known to the Committee for the determination of naphthalene in solvent naphtha can be regarded as satisfactory and suitable for issue as a standard.

All the other tests in the 1929 edition have been revised or replaced by tests in the present edition and the 1929 versions are consequently to be regarded as obsolete and no longer standard.

DEFINITIONS .

The meanings of the names applied to coal tar and its products frequently differ significantly among those who employ the names. Apart from the desirability of eliminating the resulting ambiguity, it is necessary in the present connexion that the precise meaning of the various names be defined in order that the applicability of the standard tests may be indicated with precision. The following definitions have been adopted and shall be used for the purpose of the Committee's standard tests:—

Crude Tar: A bituminous product, viscous or liquid, resulting from the destructive distillation of organic materials.

The word "tar" must always be preceded by the name of the matter from which it is produced: coal, shale, peat, vegetable matter, etc. Its mode of production should also be indicated. (Technical Dictionary of the Permanent International Association of Road Congresses.)

The following would be proper descriptions of the tars to which the present tests will normally be applied:—

"High Temperature Coal Tar (Horizontal Retort)"

"High Temperature Coal Tar (Vertical Retort)"

"High Temperature Coal Tar (Coke Oven)"

"Low Temperature Coal Tar"

"Oil Gas Tar" ("Carburetted Water-gas Tar")

Refined Tar: High temperature coal tar from which the water and more volatile oils have been removed by distillation.

Note: The International definition reads "A tar that has been distilled or cut back with the products of distillation, to the degree necessary to give a desired consistency." This corresponds more nearly to the definition for road tar below, while the Committee's definition for refined tar corresponds closely with the International definition for dehydrated tar.

Road Tar: A product prepared by treating coal tar in such a manner that it conforms to a specification which defines its applicability for some branch of road work.

Benzole Fore-runings: The first fraction distilled from crude or refined benzole or from light oil.

It contains a high proportion of constituents boiling below 75°C and is usually rich in carbon disulphide; the fore-runings from unwashed benzole are usually rich in unsaturated hydrocarbons.

Crude Benzole: A crude product extracted from coal gas, consisting essentially of benzene and its lower homologues, and having a specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of at least 0.820 (or what is equivalent, a density $\rho_{20^{\circ}\text{C}}$ of at least 0.815 g/ml).

Unsaturated hydrocarbons and sulphur compounds are usually present. Paraffins, naphthalene, phenols and pyridine bases may also be present, the last two usually in small amounts. Many crude benzoles will yield appreciable quantities of residue resulting from the oil used in their recovery.

Note: As indicated on page 117, products having a similar distillation range are obtained by the distillation of crude tar.

Crude Low Temperature Spirit: A crude product extracted from low temperature coal gases or tar and having a specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ lower than 0.820 (or what is equivalent, a density $\rho_{20^{\circ}\text{C}}$ lower than 0.815 g/ml). The product will contain higher quantities of paraffins, naphthenes and unsaturated hydrocarbons than does crude benzole.

Solvent Naphtha: A product conforming to a specification and consisting essentially of the xylenes and possibly higher homologues.

Heavy Naphtha: A fraction of light oil or of crude benzole conforming to a specification and distilling substantially between 160° and 190°C.

Light Oil: A crude fraction distilled from high temperature coal tar and containing commercial quantities of aromatic hydrocarbons which distil below about 190°C.

Phenols and bases are usually present.

Note: The term "crude naphtha," formerly reserved for light oils containing a high proportion of low boiling hydrocarbons, is becoming obsolete.

Middle Oil or Carbolic Oil: A fraction distilling substantially between 190° and 235°C, obtained from high temperature coal tar and containing commercial quantities of phenols and frequently of naphthalene.

Creosote Oil: The oil or a blend of oils obtained from coal tar and distilling above about 200°C.

The term "creosote oil" should always be associated with an

indication of the type of coal tar from which it has been produced, e.g.:—

“High Temperature Coal Tar Creosote Oil”

“Low Temperature Coal Tar Creosote Oil”

Creosote oil is usually made to a specification; consequently the oil as obtained by the distillation of coal tar generally requires suitable treatment so that it will comply with such specification requirements.

Cresylic Creosote: Creosote oil containing substantial amounts of phenols and usually conforming to a specification, especially in regard to its phenols content.

It may be obtained directly by the distillation of coal tar or it may be an oil in which the proportion of phenols has been adjusted to secure compliance with the specification.

Heavy Oil: A high boiling creosote oil obtained from coal tar and distilling above about 270°C.

Anthracene Oil: A heavy oil having a specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ above about 1.080 (or what is equivalent, a density $\rho_{20^{\circ}\text{C}}$ above about 1.075 g/ml), usually distilled from horizontal retort tar or coke oven tar.

When the expression “anthracene oil” is used, there should always be an indication whether the oil is filtered or unfiltered.

Crude Anthracene: The solid product, containing anthracene, obtained on cooling the coal tar distillate collected above about 270°C.

Coal Tar Phenols; Phenols: The mixture of those constituents of coal tar or of coal tar fractions which are soluble in aqueous caustic soda solution.

The mixture consists essentially, but not always entirely, of monohydric phenols.

Phenol: While the terms “coal tar phenols” and “phenols” have the meaning of the previous definition, the word “phenol” has been reserved throughout to indicate pure phenol, i.e. monohydroxybenzene.

Crude Carbolic Acid: A crude mixture of monohydric phenols of substantial phenol content and complying with the applicability paragraph of the test described in Serial No. C.C. 3, page 205.

Refined Carbolic Acid: A mixture of monohydric phenols

of substantial phenol content and refined to conform to a specification.

Crude Cresylic Acid: A crude mixture of monohydric phenols, essentially cresols and higher homologues, of lower phenol content than crude carbolic acid.

Refined Cresylic Acid: A refined mixture of monohydric phenols, consisting essentially of cresols and/or xylenols with or without a percentage of phenol.

Crude Naphthalene: The solid product, consisting essentially of naphthalene, obtained on cooling crude intermediate fractions from the distillation of coal tar.

Crude naphthalene is also obtained during the purification of coal gas.

Refined Naphthalene: Naphthalene commercially free from other hydrocarbons and from phenols, bases and other impurities.

Crude Tar Bases: The mixture of those constituents of coal tar which can be extracted from the lower boiling distillates by dilute mineral acid. The mixture consists essentially of basic compounds of the pyridine series.

Refined Pyridine Bases: A mixture of tar bases, consisting essentially of pyridine and its homologues, and refined to conform to a specification.

Refined Heavy Bases: A mixture of the higher boiling tar bases, refined to conform to a specification.

Pitch: The black or dark brown solid or semi-solid fusible and agglomerative residue remaining after partial evaporation or fractional distillation of tars and tar products (Technical Dictionary of the Permanent International Association of Road Congresses).

It is desirable that the derivation of the pitch be indicated, e.g.:—

“High Temperature Coal Tar Pitch”

“Low Temperature Coal Tar Pitch”

Note: Pitches resulting from the distillation of tar oils and other tar products are not generally recognized in commerce. The tests for pitch in Serial Nos P.T. 1 to 8, pages 305 to 330, are intended to apply to normal commercial pitch which would properly be defined by the omission of the words “and tar products” from the International definition.

“Free Carbon”: The solid carbonaceous matter remaining after the extraction of coal tar or pitch with pure toluole.

It is realized that so-called “free carbon” is not composed entirely of carbon and is actually a complex mixture, varying according to the solvent used. Pure toluole has been selected from among the solvents variously used, being the most suitable on practical grounds.

Tar Acids: The use of the term “tar acids” has given rise to the belief that tar products contain acids comparable with the mineral acids in their corrosive action on, for example, metals. Where this belief prevails it is difficult to dispel it by argument. The Committee has consequently adopted the term “coal tar phenols” or “phenols” for use where the term “tar acids” would otherwise be used.

SAMPLING

I. Introduction

No specification, however explicit, can take the place of judgment, skill and experience. The following directions are intended to supplement such experience and to serve as a guide in selecting the best method.

Samples are required for test purposes and, in connexion with specifications, an *average* sample is generally required. Thus, unless it be known that the bulk stock is homogeneous, steps must be taken either to mix the bulk stock or to collect a sufficient number of portions in such a way that a sample obtained by mixing these portions represents truly the average composition of the bulk.

If it be desired to check the uniformity of the product, samples taken from separate containers shall be submitted to a preliminary examination before the final blending for complete analysis.

II. General Precautions

Precautions shall be taken to ensure that the hands of the operator, the sampling apparatus, the sample containers and the samples themselves are not contaminated by extraneous substances. It is equally important that nothing in the sampling procedure should lead to contamination of the bulk material, e.g. by dirt picked up by an oily bung, by fragments of stoppers, or by other foreign matter.

When liquids are being examined prior to sampling, the presence of any separated aqueous liquor on the top or on the bottom (see Addendum A, page 29), or of frozen water or other solid matter should be reported. The result of such examination may be used to assist in deciding upon the number of packages to be actually sampled.

Several coal tar products are hygroscopic or are otherwise affected by air. Samples of these must be protected by reducing atmospheric exposure to the minimum during manipulation and by storing them in vessels which can be closed securely.

* The provisions of this section and of B.S. 616-1933: Sampling of Coal Tar and its Products are, the same.

Identical precautions shall be observed when dealing with samples of those products which tend to lose volatile constituents at atmospheric temperatures.

Samples of those products which are affected by exposure to light shall be taken and stored in metal vessels or in amber coloured or blue glass bottles (see Notes i to iv, below).

The quantities of each separate sample, whether required for reference or analysis, shall be as specified in the appropriate sections of standard tests, or alternatively at least three times the minimum necessary for the complete series of tests.

The size of the sample vessel used shall be such that it is nearly filled by the sample, sufficient free space being left to allow for expansion and to allow the contents to be mixed by shaking.

Rubber stoppers and composition corks shall not be used for closing any vessels containing samples of coal tar or its products. If sealing wax or paraffin be employed, great care shall be taken that the contents are not contaminated by the material of the seal when the vessels are opened.

Refined materials shall be protected by covers of oil-proof paper, metal foil, or other suitable impervious material *over* the stopper and top to keep moisture and dust away from the mouth of the vessel and to protect it while being handled. Such covers also prevent sealing substances, such as wax or paraffin, from getting on the lip when the seal is made or broken, and so protect the sample from contamination when it is poured out.

Notes:

(i) Apparatus used for sampling the following products should be constructed of glass or of metals or alloys which will not produce sparks on impact; amber coloured glass stoppered bottles shall be used for samples:—

Benzoles
Heavy naphthas
Rectified solvent naphtha
Toluoles
Xyloles

(ii) It is advisable to use glass apparatus for sampling the following liquids:—

Carbolic acid
Cresylic acid

(iii) Amber coloured or blue glass stoppered bottles shall be used for samples of:—

Carbolic acid
Cresylic acid
Orthocresol
Metacresol
Paracresol
Xylenols

(iv) Pyridine bases shall not be allowed to come into contact with any metal and therefore glass apparatus and amber coloured or blue glass stoppered bottles must always be used for taking and storing samples of these products.

III. Apparatus

The designs of apparatus referred to in the following paragraphs are given for guidance and are not intended to indicate that only apparatus of the form illustrated is to be used.

A. SAMPLING TUBES

1. Open Sampling Tube: The open sampling tube shall be of metal or glass (see Notes i to iv under "II. General Precautions" above) and shall be of uniform bore throughout. It shall be of such bore that when immersed the upper end can be sealed by the thumb, so that the whole of the contained liquid may be withdrawn from the bulk and discharged into the sample vessel.

2. Closable Sampling Tube: The closable sampling tube shall be constructed of metal or glass (see Notes i to iv under "II. General Precautions" above), one end being provided with a valve. Fig. 1, page 16, shews a convenient form of this apparatus constructed in metal. The construction and arrangement of the valve shall be such as will not interfere unduly with the drawing of a complete sample from the extreme top to the extreme bottom of the liquid.

When tubes are used for sampling, it is essential that they be lowered as slowly as practicable in order that the level of the liquid inside the tube shall be as nearly as possible the same as that outside, unless it be desired to take a sample at a

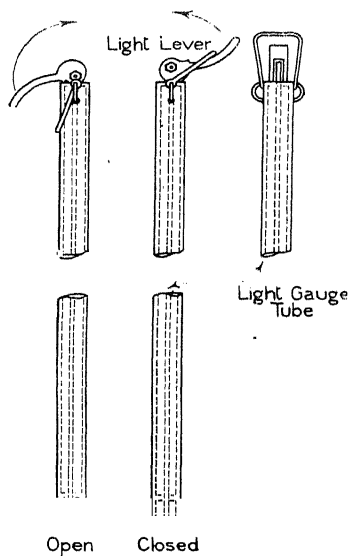


Fig. 1.—Closable Sampling Tube.

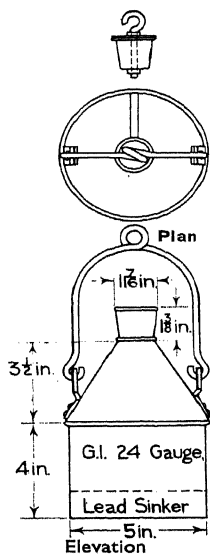


Fig. 2.—Sampling Can.

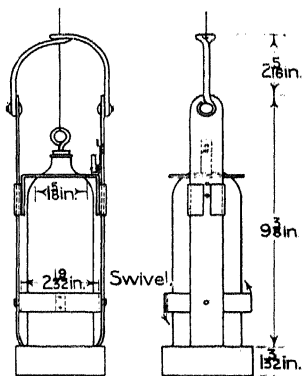


Fig. 3.—Sampling Can with Removable Bottom.

particular level. If experience has shewn that it is necessary, the tube shall be wiped externally before it is emptied, in order to prevent adherent liquid from dripping into the sample.

B. SAMPLING CAN OR BOTTLE

This apparatus consists of a weighted can or bottle attached to a length of cord or to a suitable rod, by means of which it may be lowered into a tank for sampling the liquid contents at any required level. The cork is withdrawn when the can or bottle is in the desired position. The can or bottle shall be wiped externally before the contents are discharged. Figs 2 and 3, opposite, shew suitable forms of apparatus.

C. SAMPLING SCOOP

The sampling scoop is intended for use in sampling naphthalene, anthracene, etc. and is of the type used for sampling butter. Fig. 4, shews a suitable design. The scoop shall be long enough to sample right through the package; it may be necessary to insert the implement at each end to achieve this.

D. AGITATOR

The agitator shall be of sufficient area to give adequate disturbance of the product and sufficiently light for the operator to be able to move it rapidly through the liquid. Containers differ so much in shape and size that no specific design of agitator is recommended. A form which is often suitable consists of a metal disc about 18 inches in diameter, perforated with a number of one inch holes (see Fig. 5, page 18), the disc being fixed centrally to a long metal rod or pipe, by means of which hand stirring is possible.

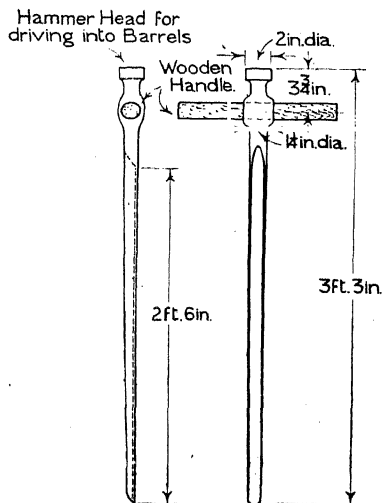


Fig. 4.—Sampling Scoop.

Note:

The Committee has not recommended the use of air for agitation as it is considered that, although it is a suitable method in many cases, its indiscriminate use might be dangerous or might lead to alteration or contamination of the material to be tested.

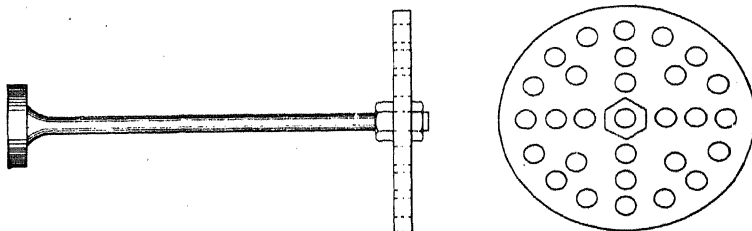


Fig. 4.—Agitator.

Perforations 1 in. in Diameter.

IV. General Procedure

A. PACKAGES (CASKS, DRUMS, SACKS, ETC.)

Wherever practicable, every container in a consignment shall be sampled, but as this is often inconvenient, the minimum number to be sampled may be determined from Table 1, below, or from Graph 1, opposite. The quantity drawn from each package shall be approximately proportionate to the contents of the package. Variations of this procedure are permissible only if they are agreed between the parties concerned.

TABLE 1: SAMPLING OF PACKAGES

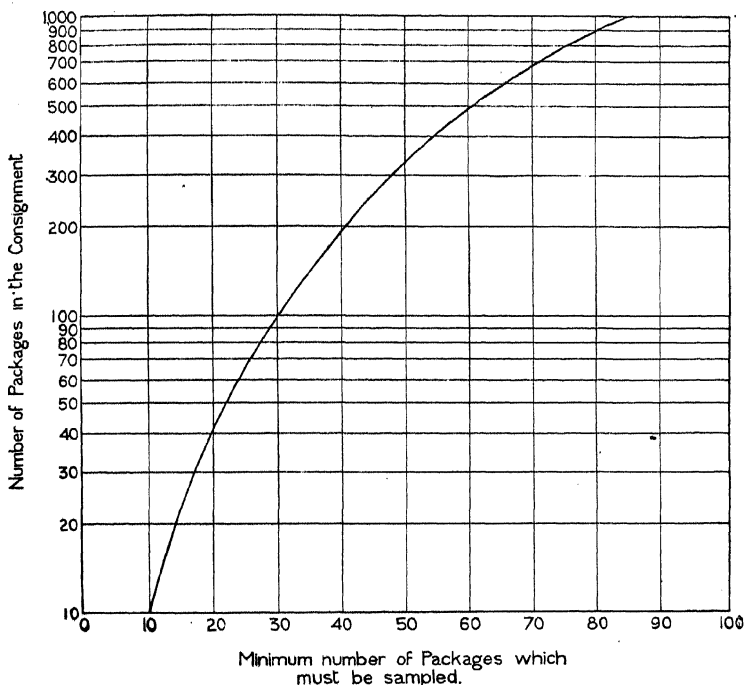
Number of packages in consignment= N	Minimum number of packages to be sampled
1 to 10	Each one
11 to 20	$10 + (N - 10)/3$
21 to 50	$14 + (N - 20)/4$
51 to 100	$22 + (N - 50)/7$
101 to 200	$30 + (N - 100)/9$
201 to 500	$41 + (N - 200)/15$
501 to 1,000	$61 + (N - 500)/22$

B. TANKS

Samples at various levels shall be taken successively downwards.

1. TANKS OF UNIFORM HORIZONTAL CROSS-SECTION
THROUGHOUT THEIR DEPTH

If the tank be not too deep, a sampling tube may be used; otherwise it is necessary to use a sampling can, withdrawing



Graph 1.—Sampling of Packages.

portions of the liquid at equally spaced levels throughout. Where the sampling is carried out by a sampling can, this should if possible be fixed on a calibrated rod so that there is no possibility of sampling at the wrong level.

In the case of large tanks where a number of manholes are available, it is recommended that the samples be taken successively downwards at each of the manholes in the roof of the tank.

2. HORIZONTAL CYLINDRICAL TANKS

Cylindrical tanks, when placed horizontally, should in general practice be sampled in accordance with Fig. 6, below, and Table 2, below, samples of equal amount being taken, one at A, two at B, two at C, and one at D. If the tank be not approximately full, the procedure shall be modified accordingly.

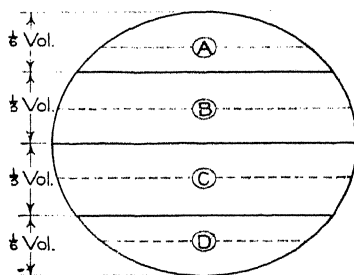


Fig. 6.—Sampling of Horizontal Cylindrical Tanks—Sampling Positions.

TABLE 2: SAMPLING OF HORIZONTAL CYLINDRICAL TANKS

Volume centres	Number of samples to be taken	Depth of sampling positions	
		Cylindrical vessels	Vessels with hemispherical ends
A	1	$d \times 0.14$	$d \times 0.18$
B	2	$d \times 0.37$	$d \times 0.39$
C	2	$d \times 0.63$	$d \times 0.61$
D	1	$d \times 0.86$	$d \times 0.82$
d is the diameter of the vessel.			

3. DISH-ENDED VESSELS

In the case of dish-ended vessels, the sampling depths shall be adjusted to the proportion of the volume of the ends to the

volume of the cylindrical portion; e.g. in the case of a boiler 6 feet in diameter, of which the volume of the ends together is equal to one-sixth of that of the cylindrical portion, the samples at A and D (see Fig. 6, opposite) should be taken about half an inch lower and higher respectively.

4. TANKS, INCLUDING TANK BARGES, DIVIDED INTO COMPARTMENTS

Every compartment shall be sampled if accessible. If any compartment be inaccessible, samples shall be drawn from the stream while charging or discharging the tank.

5. SHIP TANKS

The sampling of creosote oil and crude tar while lying in ship tanks is not recommended.

C. PIPE LINES

The sampling of heterogeneous liquids by tapping a pipe-line must always be regarded with suspicion and the practice is not to be recommended if other methods are available.

Whenever practicable, heterogeneous liquids are best sampled at the open end of the discharge pipe by means of a ladle or can. The ladle or can shall be large enough to take the whole of the contents of the stream for a short specified period, and samples shall be so taken at stated intervals of time or quantity during the whole period of loading or unloading. The samples shall be mixed and reduced to a convenient size by the method described in Section VI.B, page 24, under "Mobile Heterogeneous Liquids."

1. CONTINUOUS SAMPLING

If tapping a pipe-line be the only available method, the sampling point must if possible be situated in a rising section of the pipe-line and on the discharge side of any pump which may be installed. A small pipe shall be inserted at the chosen point and it shall extend internally one-third of the distance across the diameter of the pipe-line, the inner end being bent at 90° to face the stream. A cock shall be provided, by the adjustment of which it is possible to ensure a steady flow into the sampling vessel throughout the transfer of the liquid.

2. ALTERNATIVE METHOD OF CONTINUOUS SAMPLING

(After A.S.T.M. Standards, 1933, Part II, page 846: Methods of Sampling Petroleum and Petroleum Products (D 270-33))

Apparatus:

The continuous sampling connexion shall be constructed and installed in accordance with the form and dimensions shewn in Fig. 7, below.

The agitator shall be of the form and dimensions shewn in Fig. 5, page 18.

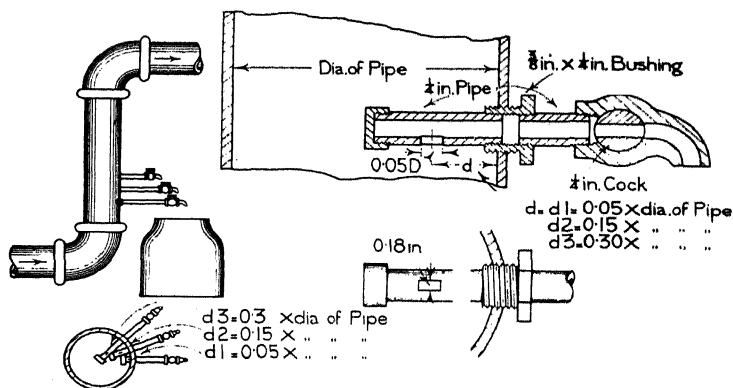


Fig. 7.—Continuous Sampling Connexion.

Method:

The three plug cocks shall be opened as nearly as possible to the same angle of opening, so that a steady stream is drawn off from each cock and at such a rate as will allow about 0.1 per cent of the stream to be diverted during the passage of the lot being sampled, but not more than 40 gallons. The following points shall be observed:—

(a) For semi-liquid materials, the receiver or receivers shall be kept warm by means of steam coils adjusted to keep the material just above the liquefying temperature.

(b) The receiver or receivers shall be kept covered and closed except for their vents and filling stream openings or pipes.

(c) Screens of larger area than the openings may be placed around the $\frac{1}{4}$ inch pipes or $\frac{1}{4}$ inch screen fittings inserted in the drip lines.

(d) A pipe cap with a suitable orifice hole may be substituted for the cocks, where the orifice size has been predetermined so as to give the right amount of sample.

(e) The gross continuous sample as collected shall be agitated with the churn dasher type of agitator, or mixed by rolling or shaking, and then sampled by means of a thief to get a one quart sample, or more where larger quantities are required.

V. Classification

The various products are classified as follows for the purpose of sampling:—

- A. Mobile homogeneous liquids
- B. Mobile heterogeneous liquids
- C. Viscous and partly solidified products
- D. Solid products

Methods are described in Section VI for drawing the bulk sample of each class of product and for preparing the final sample therefrom.

VI. Method of Drawing Bulk Sample and Preparation of Final Sample

A. MOBILE HOMOGENEOUS LIQUIDS

Bulk Sample:

Mobile homogeneous liquids may be sampled, after thorough mixing, by means of an open tube or by any of the other apparatus prescribed for liquids. Any settled aqueous layer accompanying an otherwise homogeneous liquid should if possible be drawn off before sampling the product and its quantity reported separately (see Addendum A, page 29).

If the product be contained in casks or drums, the quantity of any settled aqueous layer must be ascertained either by withdrawing and measuring it, or by emptying the vessels completely into a calibrated tank and allowing time for separation by gravity. A supernatant layer can be measured only by the second method.

If it be impracticable to apply either of the above methods, a strip of water-finding paper or paste, attached to a dipping rod of appropriate length, may be used for determining the amount

of subnatant water, provided that the coloration of the paper by the product does not mask the effect of the water.

Final Sample:

The bulk sample must be received in a vessel which is large enough to facilitate thorough mixing and it shall be agitated, preferably by shaking, immediately before withdrawing *any* portion required for analysis or for reference.

B. MOBILE HETEROGENEOUS LIQUIDS

Bulk Sample:

The mixture shall be agitated thoroughly immediately before sampling in order if possible to ensure complete mixing, and the agitation shall be continued while the samples are being drawn.

If the product be contained in drums or other small packages which are approximately full, it shall be transferred to a sufficiently large vessel before agitating and sampling.

In other respects, the procedure for this class of liquids shall follow that described for homogeneous liquids.

Final Sample:

The bulk sample shall be received in a vessel which is large enough to facilitate thorough mixing and it shall be agitated, preferably by vigorous shaking, immediately before withdrawing *any* portion required for analysis or for reference.

C. VISCOUS AND PARTLY SOLIDIFIED LIQUIDS

The products in this category include:—

1. Those crude tars and refined tars which are too viscous at the prevailing temperatures to allow of satisfactory agitation.
2. Light and middle oils, creosote and anthracene oils containing solid compounds (chiefly hydrocarbons) in suspension or as a sediment.
3. Occasionally benzoles, metacresol and carbolic acids.

If they are contained in vessels which are provided with closed steam coils or steam jackets and which are large enough to permit satisfactory agitation, the products may be warmed until they are fluid and sufficiently mobile to be treated as homogeneous liquids. Otherwise the only satisfactory practice is to draw samples at the time of loading, using one of the methods already described.

D. SOLID PRODUCTS

1. CARBOLIC ACID (PHENOL), ORTHOCRESOL, METACRESOL, PARACRESOL AND XYLENOLS

As the colour and condition of carbolic acid, orthocresol, metacresol (when solid), paracresol and xyleneols may be altered by prolonged heating and by the absorption of traces of water, the following method is recommended:—

A number of drums shall be selected from the consignment and allowed to stand in an open tank on a grid below which a closed steam coil is fitted. Immediately the material is completely melted, it shall be stirred with a clean smooth hardwood agitator and a sample drawn from each vessel by means of a glass tube, exposure to the atmosphere being minimized.

When the material is in detached form the following procedure shall be adopted:—

A sampling scoop shall be used. The scoop may be made of stainless steel and must be kept scrupulously clean, since the products to be sampled become discoloured very easily. The scoop shall be long enough to reach the bottom of the package and shall be of the partially closed type (Fig. 4, page 17). It will be advantageous if the end of the scoop is somewhat sharply pointed.

The lid of the package shall be removed and the scoop inserted and driven in with a mallet if necessary. The scoop should remain filled during withdrawal from the package. This is facilitated by inserting the scoop at an angle with the open side underneath. The scoop is given two or three turns and withdrawn with the open side uppermost.

The sample drawn in the above manner is transferred to a wide mouthed, amber coloured or blue glass stoppered bottle of suitable size. The sample which is drawn shall not be exposed to air and light longer than is absolutely necessary. The sampling shall be repeated until the bottle contains sufficient material for the purpose intended.

2. NAPHTHALENE AND ANTHRACENE

Bulk Sample:

(a) *In Sacks:* A sampling scoop shall be used and shall be thrust into the package diagonally until it meets the opposite side. The scoop shall remain filled during withdrawal from the sack. All fragments of sacking shall be removed from the sample.

(b) *In Casks:* Fresh holes shall be bored in each end, so that a scoop may be inserted and driven in with a mallet if necessary. The scoop shall remain filled during withdrawal from the package. All wood borings shall be removed from the sample.

Note:

If the product be in hard lumps, an agreed selection of the packages shall be emptied and an equal number of lumps taken from each. The separate lumps shall be trimmed to about a uniform size before combining them to form the bulk sample.

(c) *In Heaps:* The best method is to flatten the heap as far as practicable and to dig two trenches at right angles straight through it, symmetrically to the heap, the material removed in this way being regarded as the bulk sample.

Final Sample:

The bulk sample shall be reduced by quartering, which shall be done as rapidly as possible, on a clean non-absorbent surface. Instructions for quartering are described under Sub-section 4, page 28.

Notes:

(i) When a bulk sample of anthracene has been reduced by quartering to the quantity required, it shall be rubbed through a standard No. 18 test sieve (B.S. 410: Test Sieves) and then thoroughly mixed before it can be regarded as a satisfactory final sample.

(ii) If, owing to oiliness it be not possible to pass the anthracene through a No. 18 test sieve, the following procedure has been found to be satisfactory: The whole of the bulk sample is passed through a standard $\frac{3}{16}$ inch test sieve and is then reduced by coning and quartering to about 5 lb (minimum). The reduced sample is passed through a standard No. 8 test sieve. If the material be of a particularly oily nature and it be not possible to pass it through the No. 8 test sieve, the reduced sample is passed through the $\frac{3}{16}$ inch test sieve a second time.

3. PITCH

Bulk Sample:

Pieces shall be taken systematically from all parts of the material and shall be either of such size as to weigh about 200 g (about 8 oz.) each, or if larger shall be reduced to about this weight.

(a) *Pitch Shipments:*

(i) Thirty to forty pieces each about 200 g (about 8 oz.) shall be taken from the conveying plant or trucks at regular intervals throughout the loading or unloading of the pitch; or

(ii) Thirty to forty pieces each about 200 g (about 8 oz.) shall be taken from the cargo.

Ships' cargoes should be sampled during loading or unloading, as it is obvious that if sampling be postponed until loading is completed, only the material at the top of the cargoes will be represented.

(b) *Road and Rail Vehicles:*

Six pieces each about 200 g (about 8 oz.) shall be taken from each vehicle.

(c) *Packages:*

(i) *Barrels and Drums:* One end shall be removed from each of an agreed number of packages. The upper layer of pitch shall be removed to the depth of two inches and if any foreign matter be noticed, its presence shall be reported. Pieces of pitch each about 200 g (about 8 oz.) shall be cut, one from each package.

(ii) *Sacks:* An agreed selection of sacks shall be emptied and an agreed number of pieces taken from each. The separate pieces shall be trimmed to weigh about 200 g (about 8 oz.) each.

(d) *In Store (In the Bays):*

An agreed number of holes shall be made to the full depth of the pitch, the actual number of holes depending on local conditions. Pieces shall be cut out at evenly spaced levels.

It is recommended that the following number of pieces be taken from each hole, according to the thickness of the pitch:—

Up to 1 foot thick	One piece including the complete depth
Over 1 foot up to 3 feet thick	..	Two pieces
Over 3 feet up to 6 feet thick	..	Three pieces
Over 6 feet	Four pieces

Each piece shall be trimmed to weigh about 200 g (about 8 oz.).

Final Sample:

The pieces representing the bulk sample shall be mixed and spread on a clean hard surface. The bulk sample shall be

broken down until the pitch will all pass a standard $\frac{1}{4}$ inch test sieve. The broken down bulk sample shall be quartered as described under Sub-section 4, below, until it is reduced to a bulk that will conveniently provide the required number of final samples, each of which shall consist of about 1,000 g (say 2 lb).

It is recognized that the method of sampling given above takes no account of the dusty smalls which are almost invariably present in bulk consignments of pitch. If it be desired to sample this fine material, it is recommended that the procedure should be a matter for agreement between the parties concerned. Likewise, the procedure when tests are required by the buyer for any special purpose shall be a matter for agreement between the parties concerned.

4. REDUCTION OF BULK SAMPLES BY QUARTERING

The broken down sample shall be placed on a clean impervious surface and mixed by shovelling. It shall be heaped into a cone by depositing separate small quantities one on top of the other. The cone shall be formed symmetrically throughout, with its apex always in the same vertical line. A new cone shall be formed twice in a similar way, taking care to work steadily round the previous one until it is all transferred.

The third cone shall be flattened by pressing on it with a metal sheet or other suitable appliance, the flattening being carried out symmetrically and thus resulting in a mass of approximately uniform thickness and diameter. The sample shall at this stage be divided into quarters by pressing through it a metal cross constructed from four arms or blades which meet at a common centre and are held at right angles to each other by stays. A pair of opposite quarters shall be removed completely and rejected. The two remaining quarters shall be mixed together thoroughly, reduced in bulk by further quartering and the procedure repeated until a final sample of suitable quantity remains.

Alternatively, the cone may be formed with greater ease and accuracy by shovelling the material into a funnel having a short cylindrical stem about 2 inches (say 50 mm) in diameter. This funnel shall be supported in a vertical position above the top of the cone and shall be raised (without lateral displacement) as the cone grows higher, to allow the material to flow out gently and not fall from a height.

SAMPLING

Addendum A

SAMPLING OF CREOSOTE OIL WITH AN AQUEOUS LAYER AT THE BOTTOM

The following method for sampling creosote oil with a layer of water at the bottom is included for general information:—

In the case of mobile liquids existing in the form of two or more layers, such as water or emulsion below or above the oil, a representative sample of each layer is sometimes required. In such a case, although the separating line between the two layers may be more or less defined each layer may itself be heterogeneous. To attempt to stir and homogenize one or

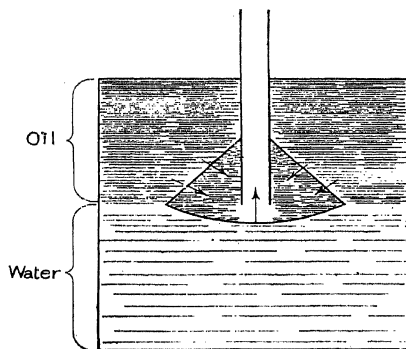


Fig. 8.—Take-off Pipe with Baffle.

each of the layers separately would result in admixing one layer with the other. To sample each layer without mixing, by means of a sampling tube or by a sampling can at various depths, might be considered insufficiently representative.

The alternative is to remove one layer from the other to a separate container, when each layer can be separately and thoroughly agitated and sampled by a sampling tube or other suitable means. The removal of one layer from another, however, is a process which in the ordinary way is extremely difficult and tedious, if a perfectly clean cut is to be obtained in a reasonable time.

Fig. 8, above, shews a device used to overcome the effects of the local suction at the end of a take-off pipe. The device comprises a slightly curved saucer-like baffle rigidly supported below, and close to, the open end of the pipe. The

diameter of the baffle is about four times that of the pipe; the distance from the bottom of the baffle to the pipe end is about one-half the diameter of the pipe; and the depth of the baffle at its centre is about one and a half times the diameter of the pipe. The pipe end fitted with this device is lowered into the bulk to be separated to such a depth that the upper edge of the curved baffle is exactly on the predetermined line of separation of the two layers. The upper layer may then be drawn off by pumping or other means of suction, at the maximum rate desired without the least disturbance of the lower layer. The movement of the liquid is entirely confined to the upper layer and follows the direction indicated by the arrows in Fig. 8, page 29.

The line of separation in the original bulk is, of course, predetermined by depth sampling, water-finding paper or other suitable means.

DENSITY AND SPECIFIC GRAVITY

In the 1929 edition of "Standard Methods," the Committee recorded its view that density (mass per unit volume), being a fundamental concept, was to be preferred to specific gravity (the ratio of the density of a substance to the density of water, the temperature of the substance and of the water being specified). The Committee's investigations at that time shewed however that, while the employment of density instead of specific gravity was increasing both in scientific and industrial work, the established custom in the tar and tar products trade was to use specific gravity as the basis of specifications and the measurement of liquids in bulk. Consequently, specific gravity tests alone appeared in the 1929 edition of "Standard Methods."

The relative merits of working on a density basis and on a specific gravity basis have since that time been admirably discussed from a detached standpoint and with special attention to the use of one or the other in specifications and in the measurement of liquids in bulk, notably in a paper "Hydrometers and Hydrometry," appearing in the Report of the World Petroleum Congress, Vol. II, page 880, London, 1933 and in B.S. 718—1936: Density Hydrometers, Appendix E of which is largely devoted to this particular point.

The question of standardizing on the basis of density or specific gravity has again been closely examined by the Committee, which decided that, as its function is to issue standard methods for the tests required for tar and tar products, provision must be made in the present edition for both density and specific gravity determinations; this provision is made in Serial No. G.P. 1, below, and Serial No. G.P. 2, page 47.

SERIAL No.: G.P. 1-38

Density

Definition:

Density is mass per unit volume.

For the purposes of the standard tests, density shall be expressed in grams per millilitre. It is recommended that whenever practicable the density at 20°C be employed. The symbol ρ_t is used to indicate the density of a substance in grams per millilitre at t°C.

I. DENSITY OF LIQUIDS

Apparatus:

A standard density bottle (Schedule No. VII, page 363) or a standard density hydrometer (Schedule No. H1, page 335). If the density bottle be intended for use in connexion with volatile liquids, it shall be provided with a close fitting cap. Schedule No. VII, page 363, provides for density bottles of the capacities shewn below with their respective maximum permissible errors:—

<i>Nominal capacity of bottle</i>	10 ml	25 ml	50 ml	100 ml
<i>Maximum permissible error</i>	\pm ml	0.004	0.006	0.008
			0.008	0.012

The size of bottle to be used will usually be determined by the amount of sample available and should be as large as circumstances will permit.

A bottle for which the National Physical Laboratory has supplied a certificate giving the actual determined capacity shall normally be used for standard practice. The maximum permissible errors in the capacity of the standard bottles are:—

4 parts in 10,000 on the nominal 10 ml bottle				
2.4 " " 10,000 " " "	25 ml	"		
1.6 " " 10,000 " " "	50 ml	"		
1.2 " " 10,000 " " "	100 ml	"		

Therefore if errors of the above magnitude in the density determination are negligible for the purpose for which the density is required, it will be sufficient to use a standard bottle known simply to be accurate within the prescribed limits and to accept the nominal capacity as the true capacity.

Schedule No. H1, page 335, provides for a comprehensive range of density hydrometers. As there indicated, the hydrometers are the British Standard hydrometers described in detail in B.S. 718—1936. That specification, in addition to describing the hydrometers fully, gives in considerable detail the manner of their use, and the operator is urged to make himself fully acquainted with those instructions, as the instructions under "Method" below are not necessarily applicable to, or adequate for, substances other than tar and tar products.

For samples of tar or tar products having densities above 1.000 g/ml, standard hydrometers adjusted for use in liquids of surface tension 35 dynes/cm shall be used.

The hydrometer may be used in an ordinary cylindrical hydrometer jar of suitable size; Table 3, below, shews the minimum dimensions of jars for the different series of standard hydrometers, together with the approximate amount of sample required for the use of a hydrometer in each series with a cylinder of the indicated size. The dimensions given in Table 3 are not put forward as proposed standard sizes for hydrometer jars, but for guidance in selecting suitable jars from sizes available.

TABLE 3: MINIMUM DIMENSIONS OF HYDROMETER JARS FOR BRITISH STANDARD DENSITY HYDROMETERS

Hydrometer series	Maximum permissible bulb diameter of hydrometer	Maximum permissible overall length of hydrometer	Hydrometer jars		Approximate volume of liquid required in cylinder*
			Minimum internal diameter	Minimum internal depth	
	mm	mm	mm	mm	ml
0.0005 \times 100	40	360	50	380	600
0.001 \times 100†	38	350	50	370	650
0.001 \times 100‡	30	350	40	370	400
0.002 \times 100†	32	340	40	360	400
0.002 \times 100‡	23	340	30	360	200
0.0005 \times 50	40	270	50	295	450
0.001 \times 50	30	260	40	285	300
0.002 \times 50†	25	250	35	275	250
0.002 \times 50‡	23	250	30	275	150
0.001 \times 25	30	195	40	220	200
0.002 \times 25	23	185	30	210	100

* The volumes shown allow for reading the highest points on the scale and for the bottom of the hydrometer being 20 mm from the bottom of the jar.
† Hydrometers with elongated, e.g. pear-shaped bulbs.
‡ Hydrometers with cylindrical bulbs.

Method:

A. DETERMINATION OF DENSITY BY MEANS OF DENSITY BOTTLE

Observations: The experimental part of the method consists in determining the weight (W_t grams) of sample required to fill the bottle at an observed temperature $t^\circ\text{C}$ (see Notes, page 37). Except in the case of light and middle oils which deposit soluble solids at 20°C and of creosote oil, $t^\circ\text{C}$ shall whenever practicable be 20°C and if an alternative working temperature be

necessary, it shall be within the range 15° to 25°C. In the case of the products excepted above, t°C shall whenever practicable be 38°C and if an alternative working temperature be necessary, it shall be within the range 36° to 40°C.

The accuracy with which the temperature must be adjusted to 20°C will depend on the coefficient of cubical expansion of the liquid of which the density is to be determined. With toluole for example, a liquid having a high coefficient of cubical expansion, a difference of 1.0 Centigrade degree from 20°C would introduce an error of 0.0009 g/ml if no allowance were made.

Calculation: 1. *Temperature of liquid adjusted to 20°C:* The calculations take their simplest form if the temperature of the liquid when the bottle is filled be 20°C.

If V_{20} = capacity of bottle in millilitres at 20°C (see Note i, page 37) and

W_{20} = weight in grams of liquid required to fill the bottle at 20°C, then

$$\rho_{20} = \frac{W_{20}}{V_{20}} + A$$

where A is a buoyancy correction obtained from Table 4, below, in which $\rho'_{20} = W_{20}/V_{20}$.

TABLE 4: BUOYANCY CORRECTIONS (A) FOR DENSITY DETERMINATIONS

Value of ρ'_{20}	Correction to give ρ_{20}
0.60 to 1.03	+ 0.0011
1.04 to 1.72	+ 0.0010

Example:

Capacity of bottle at 20°C (V_{20}) = 50.000 ml

Weight of toluole required to fill the
bottle at 20°C (W_{20}) = 43.213 g

$$\rho'_{20} = W_{20}/V_{20} = 43.213/50.000 = 0.8643$$

to the nearest unit in the fourth place of decimals. The appropriate buoyancy correction (A) from Table 4, is +0.0011. Hence the density of the toluole at 20°C is (0.8643 + 0.0011) g/ml, i.e. 0.8654 g/ml.

2. *Temperature of liquid t°C : (a) Calculation of density ρ_t :*

If V_{20} = capacity of bottle in millilitres at 20°C (see Note i, page 37), and

W_t = weight in grams of liquid required to fill the bottle at t°C, then

$$\rho_t = \frac{W_t}{V_{20}} + B$$

where B is a combined correction for buoyancy and for the change in volume of the bottle between 20°C and t°C, obtained from Table 5, below, in which $\rho'_t = W_t/V_{20}$.

TABLE 5: CORRECTIONS (B) FOR BUOYANCY AND CHANGE IN VOLUME OF BOTTLE

ρ'_t	Temperature t°C									
	14	16	18	20	22	24	26	36	38	40
0.60	12	12	11	11	11	11	10	8	8	8
0.80	12	12	11	11	10	10	10	7	7	7
1.00	12	12	11	11	10	10	9	6	6	5
1.20	12	12	11	10	10	9	8	5	4	4
1.40	12	11	11	10	9	9	8	4	3	3

NOTE: The corrections are expressed as units in the fourth place of decimals and are positive in sign.

Example:

Capacity of bottle at 20°C (V_{20}) = 50.004 ml

Weight of toluole required to fill the
bottle at 16°C (W_{16}) = 43.345 g

$$\rho'_{16} = 43.345/50.004 = 0.8668$$

to the nearest unit in the fourth place of decimals. From Table 5, the appropriate value of B for $\rho'_t = 0.8668$ and $t = 16^\circ\text{C}$ is +0.0012. Hence the density of the toluole at 16°C is (0.8668 + 0.0012) g/ml, i.e. 0.8680 g/ml.

(b) Calculation of density ρ_{20} from ρ_t :

Let β be the change in density in g/ml per Centigrade degree change in temperature. Then

$$\rho_{20} = \rho_t + \beta(t - 20)$$

The value of β for tar and tar products for which density tests are prescribed are shown in Table 6, below.

TABLE 6: CHANGE IN DENSITY (β) OF TAR AND TAR PRODUCTS WITH CHANGE IN TEMPERATURE

Product	β in g/ml per Centigrade degree	Temperature range of applicability
Crude tar	0.00065	15° to 25°C
Refined tar	0.00065	
Crude benzole	0.00090	
Light oil	0.00080	
Middle oil	0.00060	15° to 40°C
Benzole	0.00100	15° to 25°C
Toluole	0.00090	
Xylole	0.00086	
Solvent naphtha	0.00085	
Heavy naphtha	0.00080	
Benzene	0.00105	
Toluene	0.00092	
m-Xylene	0.00086	
Crude carbofic acid	0.00070	
Crude cresylic acid	0.00075	
Refined cresylic acid	0.00080	
Crude tar bases	0.00089	
Refined pyridine bases	0.00085	
Pure pyridine	0.00097	
Refined heavy bases	0.00085	
Creosote oil	0.00075	36° to 40°C
Anthracene oil	0.00070	15° to 25°C

NOTE:
The values in the second column are necessarily only mean figures, as the actual figure will vary in the case of many products from sample to sample. They may only be used over the range of temperature indicated in the third column, and then only with a clear recognition of their limitations. In case of dispute, the need to apply a correction must be avoided by carrying out the test at the specified temperature.

Example:

The value of β for toluole is 0.00090 g/ml per Centigrade degree. In the last example, the density of the sample of toluole at 16°C was 0.8680 g/ml. Hence the density of the sample at 20°C is

$$\begin{aligned}
 & 0.8680 + 0.00090 (16 - 20) \\
 & = 0.8680 - 0.0036 \\
 & = 0.8644 \text{ g/ml}
 \end{aligned}$$

Notes:

(i) The capacity (V_{20} ml) of the bottle at 20°C is

$$\frac{W_{20}(1 - \sigma/\Delta)}{(\rho_{20} - \sigma)}$$

where W_{20} is the apparent weight in air in grams of the water content at 20°C, ρ_{20} is the density in g/ml of water at 20°C, σ is the density in g/ml of the air at the time, and Δ is the density in g/ml of the weights used.

(ii) *Density of Tars by means of Density Bottle:* Many tars are too thick to allow the stopper of the density bottle to be pressed into position if the bottle be filled with tar. When this is so, the tar shall be warmed to about 60°C and poured into the weighed bottle, filling the bottle to at least 50 *per cent* of its capacity. The use of a small funnel will serve to prevent contamination of the neck of the bottle with tar. The partly filled bottle shall be allowed to stand at a temperature between 50° and 60°C for half-an-hour to permit escape of entangled air bubbles, after which it shall be allowed to cool and be weighed. Freshly boiled and cooled distilled water shall be used to fill up the bottle at the specified temperature and the weight of the bottle *plus* tar *plus* water shall be ascertained. The weight of distilled water required to fill the bottle completely at the specified temperature in the absence of tar shall also be determined.

The density of the tar, uncorrected for the buoyancy effect of the air, is given by

$$\rho'_t = \frac{W_2}{W_1 - W_3} \times \rho_t$$

where

W_1 = weight in grams of water required to fill the bottle completely at t°C,

W_2 = weight in grams of tar placed in the bottle,

W_3 = weight in grams of water required to complete the filling of the bottle at t°C after the tar has been weighed in the bottle, and

ρ_t = density of water in g/ml at t°C.

The density of water at 20°C is 0.9982 g/ml; values for other temperatures are shown in Table 7, pages 38–39.

The density of the tar, corrected for the buoyancy effect of the air, is given by $\rho_t = \rho'_t + A'$ where A' is a correction from

TABLE 7: DENSITY OF WATER
(Based on the values given in the

t°C	0-0	0-1	0-2	0-3	0-4
15	0-999 13	0-999 11	0-999 10	0-999 08	0-999 07
16	0-998 97	0-998 95	0-998 94	0-998 92	0-998 90
17	0-998 80	0-998 78	0-998 77	0-998 75	0-998 73
18	0-998 62	0-998 60	0-998 59	0-998 57	0-998 55
19	0-998 43	0-998 41	0-998 39	0-998 37	0-998 35
20	0-998 23	0-998 21	0-998 19	0-998 17	0-998 15
21	0-998 02	0-998 00	0-997 98	0-997 96	0-997 93
22	0-997 80	0-997 78	0-997 75	0-997 73	0-997 71
23	0-997 57	0-997 54	0-997 52	0-997 50	0-997 47
24	0-997 33	0-997 30	0-997 28	0-997 25	0-997 23
25	0-997 07	0-997 05	0-997 02	0-997 00	0-996 97
36	0-993 71	0-993 68	0-993 64	0-993 61	0-993 57
37	0-993 36	0-993 32	0-993 28	0-993 25	0-993 21
38	0-992 99	0-992 96	0-992 92	0-992 88	0-992 85
39	0-992 62	0-992 59	0-992 55	0-992 51	0-992 47
40	0-992 25	—	—	—	—

Table 13, page 51, the correction there given for values of $S'_{15-5^{\circ}\text{C}/15-5^{\circ}\text{C}}$ equal to ρ' being used.

The density of the tar at 20°C is $\rho_{20} = \rho_t + \beta(t - 20)$ where β is the change in density of the tar in g/ml per Centigrade degree change in temperature from Table 6, page 36.

B. DETERMINATION OF DENSITY BY MEANS OF DENSITY HYDROMETERS

General:

From the reading taken on a standard density hydrometer floating in a liquid at any convenient temperature, the density of the liquid at 20°C may be obtained by applying corrections to the observed reading for:—

- the scale error of the hydrometer at the point of reading;
- the change in volume of the hydrometer between the temperature of the liquid and the temperature of adjustment of the hydrometer (20°C);
- the difference between the surface tension of the liquid and that for which the hydrometer is adjusted; and

IN GRAMS PER MILLILITRE

International Critical Tables, Vol. III, p. 25)

0.5	0.6	0.7	0.8	0.9	t°C
0.999 05	0.999 03	0.999 02	0.999 00	0.998 99	15
0.998 89	0.998 87	0.998 85	0.998 84	0.998 82	16
0.998 71	0.998 70	0.998 68	0.998 66	0.998 64	17
0.998 53	0.998 51	0.998 49	0.998 47	0.998 45	18
0.998 33	0.998 31	0.998 29	0.998 27	0.998 25	19
0.998 13	0.998 11	0.998 09	0.998 06	0.998 04	20
0.997 91	0.997 89	0.997 87	0.997 84	0.997 82	21
0.997 68	0.997 66	0.997 64	0.997 61	0.997 59	22
0.997 45	0.997 42	0.997 40	0.997 37	0.997 35	23
0.997 20	0.997 18	0.997 15	0.997 13	0.997 10	24
0.996 94	0.996 92	0.996 89	0.996 87	0.996 84	25
0.993 54	0.993 50	0.993 46	0.993 43	0.993 39	36
0.993 18	0.993 14	0.993 10	0.993 07	0.993 03	37
0.992 81	0.992 77	0.992 74	0.992 70	0.992 66	38
0.992 44	0.992 40	0.992 36	0.992 32	0.992 28	39
—	—	—	—	—	40

(d) the change in density of the liquid between the temperature of observation and the standard temperature 20°C.

It will frequently happen that, to the degree of accuracy required for industrial purposes, the corrections under (a), (b) and (c) are negligibly small and that under (d) may be eliminated by taking readings at 20°C. Full details of the corrections are given, however, in the following pages, so as to be available if required for actual use, or alternatively to serve as an indication as to when they may be neglected without prejudice to the degree of accuracy required.

Observations:

The hydrometer and jar shall both be cleaned before use and after the hydrometer has been cleaned it shall be handled only by the top of the stem, care being taken that the fingers touch the stem only above the graduated scale.

It is more convenient to take readings at the prevailing room temperature than to bring the temperature of the sample to a specified temperature and maintain the temperature constant. The density of the sample at any specified temperature can readily be obtained from its density at any known temperature by simple calculation or by the use of tables.

The sample, in temperature equilibrium with its surroundings, shall be poured into the hydrometer jar, the sample being poured down the side of the jar to avoid the formation of air bubbles. The contents of the jar shall be stirred, again avoiding the formation of air bubbles. The hydrometer shall be inserted into the liquid, being held by the top of the stem and released when approximately in its position of equilibrium. A little experience enables one to appreciate when the hydrometer is approaching equilibrium and to release it in such a position that it rises or falls only by a small amount when released. When the hydrometer has been released the reading shall be noted approximately and then, by pressing on top of the hydrometer stem with the finger and thumb, the hydrometer shall be depressed to two or three subdivisions (about 3 to 5 mm) beyond the position of equilibrium. The top of the stem shall not be gripped by the finger and thumb but shall be allowed to rest lightly between them. On withdrawing the hand the hydrometer rises steadily and after a few oscillations settles to its position of equilibrium. The meniscus surrounding the hydrometer stem shall be observed while the hydrometer is settling to its final reading. If the stem of the hydrometer and the liquid surface are clean, the meniscus will remain unchanged as the hydrometer rises and falls. If the meniscus should crinkle or be dragged out of shape by the motion of the hydrometer, lack of cleanliness is indicated and the hydrometer stem shall be recleaned.

When the hydrometer has settled to its position of equilibrium, the scale reading corresponding to the plane of intersection of the level liquid surface and the stem shall be noted. When taking the reading, the scale shall be viewed through the liquid and with the line of sight in the plane of the level liquid surface. The hydrometer must not be touching the side of the vessel when the reading is taken.

In general, it is sufficient to observe the temperature of the liquid immediately after taking the hydrometer readings, but it is safer to take the temperature both immediately before and after the hydrometer readings, as this will ensure the detection of any unexpected excessive change.

If the liquid be too opaque for the scale to be seen through the liquid, the scale reading shall be taken where the meniscus merges into the stem of the hydrometer. The height of the top of the meniscus above the general level of the surface shall be estimated in terms of subdivisions of the hydrometer scale and

an appropriate amount added to the reading taken at the top of the meniscus to arrive at the reading corresponding to the level surface of the liquid. If the liquid be viscous as well as opaque, the hydrometer stem shall not be immersed beyond the reading, as too much liquid will adhere to the stem and the scale will be obscured. In viscous liquids the hydrometer settles only slowly to its final position of equilibrium and adequate time shall elapse for equilibrium to be attained before the reading is taken.

All hydrometers, after use at a higher temperature than 38°C, shall as a matter of precaution be allowed to drain and cool in the vertical position.

Corrections:

(a) *Correction for scale errors:* Before being put into service, all hydrometers should be tested to check the accuracy of their scales. They should be examined subsequently from time to time to see that there has been no displacement of the paper scale during use. Any displacement can be detected by lack of coincidence between the horizontal line etched on the stem and the datum line marked on the paper scale thus >—< .

For many purposes it will be sufficient to know that the scale errors are not in excess of the maximum errors specified in Schedule No. H1, page 335. For certain purposes, however, a greater degree of accuracy may be required and then the scale errors must be known. Suppose that as the result of an appropriate test it is found that a Series 0.0005 \times 100 hydrometer of range 0.850 to 0.900 reads 0.8700 g/ml in a liquid of which the temperature is 20°C, the surface tension 30 dynes/cm and the density 0.8698 g/ml. If the hydrometer be read at 20°C in a liquid having the surface tension 30 dynes/cm and the reading be in the neighbourhood of 0.870 g/ml, the correction -0.0002 g/ml must be applied to the scale reading in order to obtain the density of the liquid at 20°C.

Suppose such a hydrometer, which will be referred to as hydrometer A, to be floating side by side with a correct hydrometer B (adjusted also to indicate ρ_{20} at 20°C and for a surface tension of 30 dynes/cm) in a liquid of which the temperature is 20°C, the surface tension 30 dynes/cm and a density about 0.870 g/ml. Hydrometer A would read two-fifths of a subdivision higher than hydrometer B (one subdivision being equivalent to a change in density of 0.0005 g/ml on a Series 0.0005 \times 100 hydrometer and the correction to hydrometer A being -0.0002 g/ml) and the application of the correction

—0.0002 g/ml to the reading on hydrometer A would bring it into agreement with the reading on the correct hydrometer B. Now suppose the liquid in which the two hydrometers are floating to be heated or cooled. The readings on both hydrometers will change, but by the same amount, and so hydrometer A will still be reading two-fifths of a subdivision higher than hydrometer B. A correction of —0.0002 g/ml to the reading on hydrometer A will therefore again bring it into agreement with the reading on the correct hydrometer B. Hence the scale correction determined at 20°C may be used at any temperature. If applied to a reading on hydrometer A at any temperature $t^{\circ}\text{C}$, it will give a corrected reading equal to that which would have been obtained directly at $t^{\circ}\text{C}$ with the correct hydrometer B. The term “hydrometer reading” in all that follows, except when otherwise definitely stated, is to be taken as meaning the reading which would have been obtained under the conditions of observation on a correct hydrometer. It will therefore mean

(i) the observed reading on a hydrometer known to be accurate within the tolerances specified—the scale errors being accepted as negligibly small, or

(ii) the observed reading on a hydrometer, as corrected on account of scale error, when the accuracy desired necessitates the application of such corrections.

(b) *Temperature correction for change in volume of British Standard density hydrometer*: If the hydrometer reading be taken at a temperature $t^{\circ}\text{C}$ other than 20°C, the correction to be applied for the change in volume of the hydrometer between 20°C and $t^{\circ}\text{C}$, to obtain the value of ρ_t is obtained from Table 8, opposite. This table also shows corrections for temperatures in the neighbourhood of 38°C for use when 38°C is the specified temperature of observation.

It may be noted that if the temperature at which the hydrometer reading is taken lies within the range of temperature shewn below, the allowance for the change in volume of the hydrometer does not exceed 0.0005 g/ml:—

Hydrometer reading	Temperature range	
	$^{\circ}\text{C}$	$^{\circ}\text{C}$
0.600	—12	to 52
0.800	—4	to 44
1.000	1	to 39
1.200	4	to 36
1.400	6	to 34

(c) *Surface tension corrections for British Standard density hydrometer:* The British Standard density hydrometers have been designed to render changes in their readings due to changes in surface tension as small as practicable.

TABLE 8: CORRECTIONS FOR CHANGE IN VOLUME OF BRITISH STANDARD DENSITY HYDROMETERS

Temperature t°C	Hydrometer reading at t°C				
	0.60	0.80	1.00	1.20	1.40
14	+1	+1	+2	+2	+2
16	+1	+1	+1	+1	+1
18	0	0	+1	+1	+1
20	0	0	0	0	0
22	0	0	-1	-1	-1
24	-1	-1	-1	-1	-1
26	-1	-1	-2	-2	-2
36	-2	-3	-4	-5	-6
38	-3	-4	-5	-6	-7
40	-3	-4	-5	-6	-7

NOTES:
 (i) The corrections are expressed as units in the fourth place of decimals; when positive (+) they are to be added to and when negative (-) subtracted from the reading.
 (ii) The value 0.000026 ml per ml per Centigrade degree was used for the coefficient of cubical expansion of glass when computing the table.

The surface tensions adopted for the adjustment of the standard hydrometers have been chosen so that the surface tension corrections in liquids in which hydrometers are extensively used are quite small and, except when extreme accuracy is desired, may in general be neglected. The surface tensions of tar distillates will rarely differ by more than 5 dynes/cm from the surface tensions 20 dynes/cm to 35 dynes/cm for which the standard hydrometers are adjusted according to the range of density covered by the hydrometers. The correction due to difference in surface tensions between these liquids and the surface tensions for which the British Standard hydrometers are adjusted will not exceed one-fifth of a subdivision on the hydrometer scale and in general will be less than this amount. The surface tensions of tars may differ by slightly more than 5 dynes/cm from 35 dynes/cm, the specified surface tension for densities greater than 1.000 g/ml,

and so slightly greater errors will arise if allowance is not made for the difference.

TABLE 9: SURFACE TENSION CORRECTIONS FOR BRITISH STANDARD DENSITY HYDROMETERS

British Standard density hydrometers	Difference in surface tension (dynes/cm)	Hydrometer reading g/ml				
		0.60	0.80	1.00	1.20	1.40
Hydrometers subdivided into intervals of 0.0005 g/ml, i.e. Series 0.0005 \times 100 and 0.0005 \times 50	5	1	1	1	1	1
	10	2	2	2	2	2
	15	4	3	3	3	3
	20	5	4	4	4	3
	25	6	6	5	5	4
	30	7	7	6	5	5
	35	9	8	7	6	6
	40	10	9	8	7	7
Hydrometers subdivided into intervals of 0.001 g/ml, i.e. Series 0.001 \times 100, 0.001 \times 50 and 0.001 \times 25	5	2	2	2	2	2
	10	6	4	4	4	4
	15	8	6	6	6	6
	20	10	8	8	8	8
	25	12	12	10	10	8
	30	16	14	12	10	10
	35	18	16	14	12	12
	40	20	18	16	14	14
Hydrometers subdivided into intervals of 0.002 g/ml, i.e. Series 0.002 \times 100, 0.002 \times 50 and 0.002 \times 25	5	5	5	5	5	5
	10	10	10	10	5	5
	15	15	15	10	10	10
	20	20	20	15	15	15
	25	25	20	20	20	15
	30	25	25	25	20	20
	35	30	30	30	25	25
	40	35	35	30	30	25

NOTES:

(i) The difference in surface tension in the second column is the difference between the surface tension of the liquid in which the hydrometer is read and the surface tension for which the hydrometer is adjusted.

(ii) The corrections are expressed as units in the fourth place of decimals; the correction is to be added to or subtracted from the observed reading, according as the surface tension of the liquid under test is higher or lower than that for which the hydrometer is adjusted.

(iii) The corrections have been rounded off to the nearest 0.0001 g/ml for hydrometers subdivided in intervals of 0.0005 g/ml, to the nearest 0.0002 g/ml for hydrometers subdivided to 0.001 g/ml and to the nearest 0.0005 g/ml for hydrometers subdivided to 0.002 g/ml.

(iv) The corrections are applicable only to hydrometers conforming to B.S. 718-1936 (see Schedule No. H1, page 335, and are not applicable to density hydrometers in general.

In general, it will be possible to avoid the necessity for surface tension corrections by choosing a British Standard

hydrometer of appropriate openness of scale adjusted either for liquids with low or for liquids with high surface tensions, as may be required, in accordance with Table 30, page 338.

Table 9, opposite, has, however, been prepared to provide for circumstances where the accuracy required, or the use of the hydrometers in liquids other than tar or tar products, involves corrections for surface tension. The surface tension corrections shewn in Table 9 are applicable only to hydrometers conforming to B.S. 718—1936 (see Schedule No. H1, page 335).

(d) *Reduction of corrected hydrometer reading to density ρ_{20}* : The observed hydrometer reading corrected in accordance with (a), (b) and (c) above is the density ρ_t , $t^\circ\text{C}$ being the temperature of the liquid when the hydrometer reading is taken. If the density be required at 20°C , the standard temperature of reference, an allowance must be made for the change in density of the liquid with change in temperature. This may be done by means of the equation

$$\rho_{20} = \rho_t + \beta(t - 20)$$

where β is the change in density of the liquid in g/ml per Centigrade degree change in temperature. Values of β for tar and tar products are given in Table 6, page 36.

Example:

The determination of the density ρ_{20} of a sample of toluole by means of a British Standard hydrometer of Series 0.0005 \times 100 of range 0.850 g/ml to 0.900 g/ml and adjusted for the surface tension 30 dynes/cm will be taken as an example. It will be assumed that the correction to the hydrometer for scale error is $+0.0004$ g/ml, i.e. that the hydrometer has the maximum error permissible under the specification. The surface tension of toluole is taken as that of toluene, viz 28 dynes/cm.

Reading observed on the hydrometer in

the sample	0.8675 g/ml
Temperature of sample	15.7°C

The density $\rho_{15.7}$ of the toluole is obtained as follows:—

Observed hydrometer reading at 15.7°C	0.867 5 g/ml
Correction for scale error of hydrometer..	$+0.000 4$ g/ml
Correction for change in volume of hydrometer from Table 8	$+0.000 1$ g/ml
Surface tension correction from Table 9..	$0.000 0$ g/ml
Density $\rho_{15.7}$ of the toluole	0.868 0 g/ml

Note that if a value accurate within ± 0.001 g/ml be adequate, all corrections, including those for scale errors of the hydrometer, could be neglected and the observed reading accepted as the density of the sample at 15.7°C . If a value accurate to within ± 0.0005 g/ml be adequate, the temperature and surface tension corrections could safely be neglected, but it would be advisable to apply corrections for scale errors if these amounted to the maximum permissible (± 0.0004 g/ml) as they would then form a large fraction of the error permissible in the final result.

The value of β , i.e. the change in density in g/ml per Centigrade degree, for toluole is 0.00090. Hence, since

$$\rho_{20} = \rho_t + \beta(t - 20),$$

the density ρ_{20} of the toluole is

$$\begin{aligned} & 0.8680 + 0.00090(15.7 - 20) \\ &= 0.8680 - 0.0039 \\ &= 0.8641 \text{ g/ml} \end{aligned}$$

II. DENSITY OF SOLIDS

Pitch is the only solid tar product for which a test for density is prescribed. The sample on which the test will be performed will consist of material which will pass a $\frac{1}{4}$ inch mesh sieve as required by the specified procedure for the sampling of pitch (see page 26). The method given below has been formulated accordingly.

Apparatus:

A standard density bottle having a nominal capacity of 100 ml (Schedule No. V11, page 363). The internal diameter at the top of the neck shall not be less than 12.5 mm, the figure recommended in the specification.

Method:

The weight of the bottle shall be determined and a representative portion (not less than 50 g) of the sample shall be introduced. The weight of the bottle *plus* sample shall be ascertained. Freshly boiled and cooled distilled water shall be used to fill up the bottle at the specified temperature, precautions being taken that any entangled air bubbles are removed before the final adjustment, and the weight of the bottle *plus* pitch *plus* water shall be ascertained. The weight of distilled water required to fill the bottle completely at the specified temperature in the absence of pitch shall also be ascertained.

The density of the pitch shall be obtained from the resulting data, the calculation and corrections being as given in Note ii on page 37.

SERIAL No.: G.P. 2-38

Specific Gravity

Definition:

The specific gravity of a substance is the ratio

$$\frac{\text{Density of the substance at the temperature } t_1}{\text{Density of water at the temperature } t_2}$$

It is necessary always to specify t_1 and t_2 when referring to specific gravity and the symbol S_{t_1/t_2} is used to indicate the ratio defined above.

In the absence of other instructions the temperature 15.5°C shall be employed for both t_1 and t_2 in tar and tar products technology, i.e. specific gravities represented by the symbol $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$ shall be used, except that, for light and middle oils which deposit soluble solids at 15.5°C and for creosote oil, the specific gravity $S_{38^\circ\text{C}/20^\circ\text{C}}$ shall be employed.

Conversion of Specific Gravity to Density:

From the definition of specific gravity it follows that

$$\rho_{t_1} = S_{t_1/t_2} \times \rho_{t_2}$$

where ρ_{t_1} is the density of the substance at the temperature t_1 and ρ_{t_2} is the density of water at the temperature t_2 .

Since the density of water at 15.5°C is 0.99905 g/ml ,

$$\rho_{15.5^\circ\text{C}} = S_{15.5^\circ\text{C}/15.5^\circ\text{C}} \times 0.99905$$

The density of water at 20°C is 0.99823 g/ml and hence

$$\rho_{38^\circ\text{C}} = S_{38^\circ\text{C}/20^\circ\text{C}} \times 0.99823$$

Table 10, page 48, shows corrections, based on the above relations, for converting specific gravities $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$ into densities in g/ml at 15.5°C and for converting specific gravities $S_{38^\circ\text{C}/20^\circ\text{C}}$ into densities in g/ml at 38°C .

Example:

Determined specific gravity $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$	=	0.824 5
Correction from Table 10	=	- 0.000 8

Density of substance at 15.5°C	=	<u>0.823 7 g/ml</u>
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The density in g/ml at 20°C of a tar or tar product of known density at 15.5°C is given by

$$\begin{aligned}\rho_{20} &= \rho_{15.5} + \beta(15.5 - 20) \\ &= \rho_{15.5} - 4.5\beta\end{aligned}$$

where β is the change in density in g/ml per Centigrade degree change in temperature. Values of β are given in Table 6, page 36.

TABLE 10: CONVERSION OF SPECIFIC GRAVITY TO DENSITY

Specific gravity $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$	Correction to give density $\rho_{15.5^\circ\text{C}}$ g/ml	Specific gravity $S_{38^\circ\text{C}/20^\circ\text{C}}$	Correction to give density $\rho_{38^\circ\text{C}}$ g/ml
0.60	- 6	0.60	-11
0.65	- 6	0.65	-12
0.70	- 7	0.70	-12
0.75	- 7	0.75	-13
0.80	- 8	0.80	-14
0.85	- 8	0.85	-15
0.90	- 9	0.90	-16
0.95	- 9	0.95	-17
1.00	-10	1.00	-18
1.05	-10	1.05	-19
1.10	-10	1.10	-19
1.15	-11	1.15	-20
1.20	-11	1.20	-21
1.25	-12	1.25	-22
1.30	-12	1.30	-23

NOTE: The corrections are expressed as units in the fourth place of decimals.

Conversion of Specific Gravity Hydrometer Readings to Density Hydrometer Readings:

Consider two hydrometers, one adjusted to indicate specific gravity $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$ when read at 15.5°C in a liquid having a specified surface tension and the other adjusted to indicate density in g/ml when read at 20°C in a liquid having the same surface tension. Suppose the two hydrometers to be floating side by side in a liquid having any temperature $t^\circ\text{C}$ and the specified surface tension. Then the correction to be applied to the reading obtained on the specific gravity hydro-

meter in order to obtain a reading equal to that on the density hydrometer may be obtained from Table 11, below.

TABLE 11: CONVERSION OF READINGS ON SPECIFIC GRAVITY HYDROMETERS ($S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ at 15.5°C) TO READINGS ON DENSITY HYDROMETERS (g/ml at 20°C)

Reading on specific gravity ($S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ at 15.5°C) hydrometer	Correction to give reading on density (g/ml at 20°C) hydrometer
0.60	— 6
0.65	— 7
0.70	— 7
0.75	— 8
0.80	— 9
0.85	— 9
0.90	—10
0.95	—10
1.00	—11
1.05	—11
1.10	—12
1.15	—12
1.20	—13
1.25	—13
1.30	—14

NOTES:
 (i) The corrections are expressed as units in the fourth place of decimals.
 (ii) The above table should be used with care and bearing in mind the precise significance of the corrections as explained in the paragraph preceding the table.

1. SPECIFIC GRAVITY OF LIQUIDS

Apparatus:

A specific gravity bottle or flask, pycnometer, standard density bottle (Schedule No. V11, page 337) or standard specific gravity hydrometer (Schedule No. H2, page 363). A cap shall be fitted to any bottle used for volatile liquids.

The hydrometer may be used in an ordinary cylindrical hydrometer jar of suitable size; Table 12, page 50, gives the minimum dimensions of jars for the different series of standard hydrometers, together with the approximate amount of sample

required for the use of a hydrometer in each series with a cylinder of the indicated size. The dimensions given in Table 12 are not put forward as proposed standards but for guidance in selecting suitable jars from sizes available.

TABLE 12: MINIMUM DIMENSIONS OF HYDROMETER JARS FOR S.T.P.T.C. SPECIFIC GRAVITY HYDROMETERS

Hydro- meter series	Maximum permis- sible bulb diameter of hydro- meter	Maximum permis- sible overall length of hydrometer	Hydrometer jars		Approximate volume of liquid required in cylinder*
			Minimum internal diameter	Minimum internal depth	
A	mm 40	mm 380	mm 50	mm 380	ml 600
B	30	250	40	275	250
C	25	195	35	220	180
D	20	150	30	175	100

* The volumes shown allow for reading the highest points on the scale and for the bottom of the hydrometer being 20 mm from the bottom of the jar.

Method:

A. DETERMINATION OF SPECIFIC GRAVITY $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ BY MEANS OF A SPECIFIC GRAVITY BOTTLE

Observations: When a specific gravity bottle or flask, pycnometer or density bottle is used, the experimental part of the method consists in determining the weight of water (W_w grams) required to fill the apparatus at 15.5°C and the weight of liquid (W_L grams) required to fill the apparatus at an observed temperature $t^{\circ}\text{C}$ (see Note i, page 53). Whenever practicable, $t^{\circ}\text{C}$ shall be 15.5°C and when an alternative working temperature is necessary it shall be within the range 15.5°C to 25°C (but see Note ii, page 55).

The accuracy with which the temperature must be adjusted to 15.5°C will depend upon the coefficient of cubical expansion of the liquid of which the specific gravity is to be determined. With toluole, for example, a liquid having a high coefficient of cubical expansion, a difference of 1.0 Centigrade degree from 15.5°C would introduce an error of 0.00090 in the specific gravity if no allowance were made.

Calculation: 1. *Temperature of liquid adjusted to 15.5°C :* The calculations take their simplest form if the temperature of the liquid when the bottle is filled be 15.5°C .

If

W_W = weight in grams of water required to fill the bottle at 15.5°C. and

W_L = weight in grams of liquid required to fill the bottle at 15.5°C, then

$$S_{15.5^\circ\text{C}/15.5^\circ\text{C}} = \frac{W_L}{W_W} + A'$$

where A' is a buoyancy correction obtained from Table 13, below, in which $S'_{15.5^\circ\text{C}/15.5^\circ\text{C}} = W_L/W_W$.

TABLE 13: BUOYANCY CORRECTIONS (A') FOR SPECIFIC GRAVITY DETERMINATIONS

Value of $S'_{15.5^\circ\text{C}/15.5^\circ\text{C}}$	Correction to give $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$
0.60	+5
0.65	+4
0.70	+4
0.75	+3
0.80	+2
0.85	+2
0.90	+1
0.95	+1
1.00	0
1.05	-1
1.10	-1
1.15	-2
1.20	-2
1.25	-3
1.30	-4

NOTE: The corrections are expressed as units in the fourth place of decimals; when positive (+) they are to be added to, and when negative (-) subtracted from the value of $S'_{15.5^\circ\text{C}/15.5^\circ\text{C}}$.

Example:

Weight of water required to fill the bottle
at 15.5°C (W_W) = 49.892 g

Weight of a sample of toluole required to
fill the bottle at 15.5°C (W_L) = 43.361 g

(The above numerical value for W_W is typical of those which would be obtained with a British Standard density bottle of nominal capacity 50 ml at 20°C).

$$S'_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}} = W_L/W_W = 43.361/49.892 = 0.8691$$

to the nearest unit in the fourth place of decimals. The appropriate buoyancy correction (A') from Table 13 is $+0.0002$. Hence the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the toluole is $0.8691 + 0.0002$, i.e. 0.8693.

TABLE 14: CORRECTION (B') FOR BUOYANCY AND CHANGE IN VOLUME OF BOTTLE

$S'_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$	Temperature $t^{\circ}\text{C}$										
	14	15.5	16	18	20	22	24	26	36	38	40
0.60	+5	+5	+5	+4	+4	+4	+3	+3	+2	+1	+1
0.65	+5	+4	+4	+4	+3	+3	+3	+2	+1	0	0
0.70	+4	+4	+4	+3	+3	+2	+2	+2	0	-1	-1
0.75	+3	+3	+3	+3	+2	+2	+1	+1	-1	-1	-2
0.80	+3	+2	+2	+2	+1	+1	+1	0	-2	-2	-3
0.85	+2	+2	+2	+1	+1	0	0	-1	-3	-3	-4
0.90	+2	+1	+1	+1	0	0	-1	-1	-4	-4	-5
0.95	+1	+1	0	0	-1	-1	-2	-2	-4	-5	-5
1.00	0	0	0	-1	-1	-2	-2	-3	-5	-6	-6
1.05	0	-1	-1	-1	-2	-2	-3	-3	-6	-7	-7
1.10	-1	-1	-1	-2	-2	-3	-4	-4	-7	-8	-8
1.15	-1	-2	-2	-3	-3	-4	-4	-5	-8	-9	-9
1.20	-2	-2	-3	-3	-4	-4	-5	-6	-9	-9	-10
1.25	-3	-3	-3	-4	-4	-5	-6	-6	-10	-10	-11
1.30	-3	-4	-4	-4	-5	-6	-6	-7	-11	-11	-12

NOTE: The corrections are expressed as units in the fourth place of decimals; when positive (+) they are to be added to, and when negative (-) subtracted from the value of $S'_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$.

2. *Temperature of liquid $t^{\circ}\text{C}$: (a) Calculation of $S'_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$:*

If W_W = weight in grams of water required to fill the bottle at 15.5°C and

${}_tW_L$ = weight in grams of liquid required to fill the bottle at $t^{\circ}\text{C}$, then

$$S'_{t^{\circ}\text{C}/15.5^{\circ}\text{C}} = \frac{{}_tW_L}{W_W} + B'$$

where B' is a combined correction for buoyancy and for the change in volume of the bottle between 15.5°C and $t^{\circ}\text{C}$ obtained from Table 14, above, in which $S'_{t^{\circ}\text{C}/15.5^{\circ}\text{C}} = {}_tW_L/W_W$.

Example:

Weight of water required to fill the
bottle at 15.5°C (W_W) = 49.892 g

Weight of a sample of toluole required to
fill the bottle at 17.6°C (t_{W_L}) = 43.271 g

$$S'_{t^{\circ}C/15.5^{\circ}C} = t_{W_L}/W_W = 43.271/49.892 = 0.8673$$

to the nearest unit in the fourth decimal place. From Table 14, the appropriate value of B' for $S'_{17.6^{\circ}C/15.5^{\circ}C}$ is +0.0001. Hence the specific gravity $S_{17.6^{\circ}C/15.5^{\circ}C}$ of the toluole is 0.8673 + 0.0001, i.e. 0.8674.

(b) *Calculation of $S_{15.5^{\circ}C/15.5^{\circ}C}$ from $S_{t^{\circ}C/15.5^{\circ}C}$:*

Let β be the change in density in g/ml per Centigrade degree change in temperature. Then

$$S_{15.5^{\circ}C/15.5^{\circ}C} = S_{t^{\circ}C/15.5^{\circ}C} + \beta(t - 15.5)$$

The values for β for tar and tar products for which specific gravity tests are prescribed are shewn in Table 6, page 36.

Example:

The value of β for toluole is 0.00090 per Centigrade degree. In the last example, the specific gravity $S_{17.6^{\circ}C/15.5^{\circ}C}$ of the toluole was 0.8674. Hence the specific gravity $S_{15.5^{\circ}C/15.5^{\circ}C}$ of the toluole is

$$\begin{aligned} & 0.8674 + 0.00090 (17.6 - 15.5) \\ & = 0.8674 + 0.0019 \\ & = 0.8693 \end{aligned}$$

Notes:

(i) *Specific gravity of tar:* Many tars are too thick to allow the stopper of the specific gravity bottle or density bottle to be pressed into position if the bottle be filled with tar. When this is so, the tar shall be warmed to about 60°C and poured into the weighed bottle, filling the bottle to at least 50 per cent of its capacity. The use of a small funnel will serve to prevent contamination of the neck of the bottle by tar. The partly filled bottle shall be allowed to stand at a temperature between 50° and 60°C for half an hour to permit escape of entangled air bubbles, after which it shall be allowed to cool and be weighed. Freshly boiled and cooled distilled water shall be used to fill up the bottle at the specified temperature and the weight of the bottle *plus* tar *plus* water shall be ascertained.

The weight of distilled water required to fill the bottle completely at the specified temperature in the absence of tar shall also be ascertained.

If the determination be made at 15.5°C and

W_1 = weight in grams of water required to fill the bottle completely at 15.5°C,

W_2 = weight in grams of tar placed in the bottle and

W_3 = weight in grams of water required to complete the filling of the bottle at 15.5°C after the tar has been weighed into the bottle, then

$$S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}} = \frac{W_2}{W_1 - W_3} + A'$$

where A' is a correction for the buoyancy effect of the air, the value of $W_2/(W_1 - W_3)$ giving the appropriate value of $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ for obtaining the required correction from Table 13, page 51.

If the determination be made at some temperature $t^{\circ}\text{C}$ other than 15.5°C and

${}_tW_1$ = weight in grams of water required to fill the bottle completely at $t^{\circ}\text{C}$;

W_2 = weight in grams of tar placed in the bottle; and

${}_tW_3$ = weight in grams of water required to complete the filling of the bottle at $t^{\circ}\text{C}$ after the tar has been weighed in the bottle, then

$$S_{t/t} = \frac{W_2}{{}_tW_1 - {}_tW_3} + A'$$

where A' is the correction obtained from Table 13, page 51, as before.

The specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ is

$$\frac{1}{w\rho_{15.5}} [S_{t/t} \times {}_w\rho_t + \beta(t - 15.5)]$$

where

$w\rho_{15.5}$ = density of water in g/ml at 15.5°C;

${}_w\rho_t$ = density of water in g/ml at $t^{\circ}\text{C}$; and

β = change in density of the tar in g/ml per Centigrade degree change in temperature.

Values of the density of water at various temperatures are given in Table 7, pages 38 and 39, and values of β in Table 6, page 36.

(ii) *Specific gravity of creosote oil, etc.*: The specific gravity of light and middle oils which deposit soluble solids at 15.5°C and of creosote oil shall be determined at 38°C and the result recorded as $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$.

If W_w = weight in grams of water required to fill the bottle at 38°C; and

W_o = weight in grams of oil required to fill the bottle at 38°C, then

$$S_{38^{\circ}\text{C}/38^{\circ}\text{C}} = \frac{W_o}{W_w} + A'$$

where A' is the buoyancy correction obtained from Table 13, page 51, as before.

The specific gravity $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$ is

$$S_{38^{\circ}\text{C}/38^{\circ}\text{C}} \times \frac{\rho_{38}}{\rho_{20}}$$

where ρ_{38} is the density of water in g/ml at 38°C and ρ_{20} is the density of water in g/ml at 20°C. Substituting numerical values for these densities the following relation is obtained

$$S_{38^{\circ}\text{C}/20^{\circ}\text{C}} = S_{38^{\circ}\text{C}/38^{\circ}\text{C}} \times 0.9948$$

As indicated, the determination of the specific gravity $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$ is to be regarded as standard in this country. The use of specific gravity instead of density as a criterion of the quality of creosote oil has caused much divergence in practice; thus, while B.S. 144—1936: Coal Tar Creosote for the Preservation of Timber prescribes the specific gravity $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$ (thereby replacing the 1921 basis $S_{38^{\circ}\text{C}/38^{\circ}\text{C}}$) and gives in addition, the densities in g/ml at 20°C corresponding to the specified specific gravities $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$, the specification of the American Railway Engineering Association (which is the same as that of the American Wood Preservers' Association) is based on $S_{38^{\circ}\text{C}/15.5^{\circ}\text{C}}$; the Scandinavian specification for creosote oil and that known as the Budapest specification are based on $S_{38^{\circ}\text{C}/4^{\circ}\text{C}}$ (equivalent to density in g/ml at 38°C) and the German specification for creosote oil is based on $S_{20^{\circ}\text{C}/20^{\circ}\text{C}}$. The operator who is required to carry out tests on one or more of these alternative bases will require to modify the foregoing instructions appropriately and to take special precautions that the basis of the results reported is clearly indicated on every occasion.

A bottle having a capacity of 50.142 ml \pm 0.008 at 38°C is a convenient instrument for the determination of the specific

gravity $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$, as the weight in grams of creosote oil at 38°C required to fill such a bottle at 38°C gives $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$ when divided by 50.

It may be noted from the last equation above that if by reason of convenience or otherwise the specific gravity $S_{38^{\circ}\text{C}/38^{\circ}\text{C}}$ be determined (e.g. with a specific gravity bottle having a capacity of $50.406 \text{ ml} \pm 0.008$ at 38°C , or, under part B below, with a specific gravity hydrometer standardized at 38°C), the result may be converted to $S_{38^{\circ}\text{C}/20^{\circ}\text{C}}$ by applying the factor 0.9948 or, for all practical purposes, 0.995.

**B. DETERMINATION OF SPECIFIC GRAVITY $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$
BY MEANS OF A SPECIFIC GRAVITY HYDROMETER**

General:

The determination of the specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, by means of a standard specific gravity hydrometer involves (1) reading the hydrometer in the liquid at a known temperature and (2) applying corrections to the observed reading for:—

- (a) the scale error of the hydrometer at the point of reading;
- (b) the change in volume of the hydrometer between the temperature of the liquid and the standard temperature of adjustment of the hydrometer (15.5°C);
- (c) the difference between the surface tension of the liquid and that for which the hydrometer is adjusted; and
- (d) the change in density of the liquid between the temperature of observation and the standard temperature 15.5°C .


It will frequently happen that, to the degree of accuracy required for industrial purposes, the corrections under (a), (b) and (c) are negligibly small and that under (d) may be eliminated by taking readings at 15.5°C . Full details of the corrections are given, however, in the following pages, so as to be available if required for actual use, or alternatively to serve as an indication as to when they may be neglected without prejudice to the degree of accuracy required.

Observations:

As stated on page 49, the determination of the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of a liquid by means of a specific gravity hydrometer shall be carried out with one or other of the hydrometers of the four series specified in Schedule No. H2, page 337. Alternatively, British Standard density hydrometers may be used in conjunction with Table 10, page 48.

The method to be followed when taking readings is the same as that described on pages 39 to 41 for density hydrometers.

Corrections:

(a) *Correction for scale errors:* Before being put into service, all hydrometers should be tested to check the accuracy of their scales. They should be examined subsequently from time to time to see that there has been no displacement of the paper scale during use. Any displacement can be detected by lack of coincidence between the horizontal line etched on the stem and the datum line marked on the paper scale thus .

For many purposes it will be sufficient to know that the scale errors are not in excess of the maximum errors specified in Schedule No. H2, page 337. For certain purposes, however, a greater degree of accuracy may be required and then the scale errors must be known. Suppose that as the result of an appropriate test it is found that a Series A hydrometer of range 0.850 to 0.900 reads 0.8700 in a liquid of which the temperature is 15.5°C , the surface tension 30 dynes/cm and the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ 0.8698. If the hydrometer be read at 15.5°C in a liquid having the surface tension 30 dynes/cm and the reading be in the neighbourhood of 0.870, the correction -0.0002 must be applied to the scale reading in order to obtain the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the liquid.

Suppose such a hydrometer, which will be referred to as hydrometer A, to be floating side by side with a correct hydrometer B (adjusted also to indicate $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ at 15.5°C and for a surface tension of 30 dynes/cm) in a liquid of which the temperature is 15.5°C , the surface tension 30 dynes/cm and a specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ about 0.870. Hydrometer A would read two-fifths of a subdivision higher than hydrometer B (one subdivision being equivalent to a change in specific gravity of 0.0005 on a series A hydrometer and the correction to hydrometer A being -0.0002) and the application of the correction -0.0002 to the reading on hydrometer A would bring it into agreement with the reading on the correct hydrometer B. Now suppose the liquid in which the two hydrometers are floating to be heated or cooled. The readings on both hydrometers will change, but by the same amount, and so hydrometer A will still be reading two-fifths of a subdivision higher than hydrometer B. A correction of -0.0002

to the reading on hydrometer A will therefore again bring it into agreement with the reading on the correct hydrometer B. Hence the scale correction determined at 15.5°C may be used at any temperature. If applied to a reading on hydrometer A at any temperature $t^{\circ}\text{C}$, it will give a corrected reading equal to that which would have been obtained directly at $t^{\circ}\text{C}$ with the correct hydrometer B. The term "hydrometer reading" in all that follows, except when otherwise definitely stated, is to be taken as meaning the reading which would have been obtained under the conditions of observation on a correct hydrometer. It will therefore mean

- (i) the observed reading on a hydrometer known to be accurate within the tolerances specified—the scale errors being accepted as negligibly small, or
- (ii) the observed reading on a hydrometer, as corrected on account of scale error, when the accuracy desired necessitates the application of such corrections.

(b) *Temperature correction for change in volume of S.T.P.T.C. standard hydrometer:* The hydrometer reading, if not taken actually at 15.5°C , should be taken within the temperature range 15° to 25°C . If the hydrometer reading be taken at a temperature $t^{\circ}\text{C}$ within this range, the correction to be applied for the change in volume of the hydrometer when obtaining the specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ is obtained from Table 15, page 59. This table also shews corrections for temperatures in the neighbourhood of 38°C for use when 38°C is the specified temperature of observation.

(c) *Surface tension corrections for S.T.P.T.C. standard specific gravity hydrometer:* The surface tensions adopted for the adjustment of S.T.P.T.C. specific gravity hydrometers are appropriate for tars and tar products. In general, it will be possible to avoid the necessity for surface tension corrections by choosing a hydrometer of appropriate openness of scale.

Table 16, page 60, has, however, been prepared to provide for circumstances where the accuracy required, or the use of the hydrometers in liquids other than tar or tar products, involves corrections for surface tension. The surface tension corrections given in Table 16 are applicable only to hydrometers conforming to Schedule No. H2, page 337.

(d) *Reduction of corrected hydrometer reading to specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$:* The observed hydrometer reading corrected in accordance with subsections (a), (b) and (c) above is the

specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the liquid, $t^{\circ}\text{C}$ being the temperature of the liquid when the hydrometer reading is taken.

TABLE 15: CORRECTIONS FOR CHANGE IN VOLUME OF S.T.P.T.C. STANDARD SPECIFIC GRAVITY HYDROMETERS

Temperature $t^{\circ}\text{C}$	Hydrometer reading at $t^{\circ}\text{C}$				
	0.60	0.80	1.00	1.20	1.40
14	0	0	0	0	+1
15.5	0	0	0	0	0
16	0	0	0	0	0
18	0	-1	-1	-1	-1
20	-1	-1	-1	-1	-2
22	-1	-1	-2	-2	-2
24	-1	-2	-2	-3	-3
26	-2	-2	-3	-3	-4
36	-3	-4	-5	-6	-7
38	-4	-5	-6	-7	-8
40	-4	-5	-6	-8	-9

NOTES:
 (i) The corrections are expressed as units in the fourth place of decimals; when positive (+) they are to be added to, and when negative (-) subtracted from the hydrometer reading.
 (ii) The value 0.000026 ml per ml per Centigrade degree was used for the coefficient of cubical expansion of glass when computing the table.

The specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the liquid is given by the relation

$$S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}} = S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}} + \beta(t - 15.5)$$

where β is the change in density of the liquid in g/ml per Centigrade degree change in temperature. Values of β for tar and tar products are given in Table 6, page 36.

Example:

The determination of the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of a sample of toluole by means of a Series A S.T.P.T.C. specific gravity hydrometer of range 0.850 to 0.900, and adjusted for the surface tension 30 dynes/cm, will be taken as an example. It will be assumed that the correction to the hydrometer for scale error is +0.0004, i.e. that the hydrometer has the maximum error permissible under the specification given in Schedule No. H2, page 337. The surface tension of toluole is taken as that of toluene, viz 28 dynes/cm.

STANDARD METHODS

TABLE 16: SURFACE TENSION CORRECTIONS FOR S.T.P.T.C. SPECIFIC GRAVITY HYDROMETERS

S.T.P.T.C. hydrometers	Difference in surface tensions (dynes/cm)	Hydrometer reading				
		0-65	0-80	1-00	1-20	1-30
Series A	5	1	1	1	1	1
	10	2	2	2	2	2
	15	4	3	3	3	3
	20	5	4	4	4	4
	25	6	5	5	5	4
	30	7	7	6	5	5
	35	8	8	7	6	6
	40	9	9	8	7	7
Series B	5	2	2	2	2	2
	10	4	4	4	4	4
	15	6	6	6	6	4
	20	8	8	8	6	6
	25	12	10	10	8	8
	30	14	12	12	10	10
	35	16	14	12	12	12
	40	18	16	14	14	14
Series C	5	5	5	5	5	5
	10	10	5	5	5	5
	15	10	10	10	10	10
	20	15	15	15	10	10
	25	20	20	15	15	15
	30	25	20	20	20	15
	35	25	25	20	20	20
	40	30	30	25	25	25
Series D	5	5	5	5	5	5
	10	15	15	10	10	10
	15	20	20	15	15	15
	20	30	25	25	20	20
	25	35	30	30	25	25
	30	40	40	35	30	30
	35	50	45	40	35	35
	40	55	50	45	40	40

NOTES:

(i) The difference in surface tension in the second column is the difference between the surface tension of the liquid in which the hydrometer is read and the surface tension for which the hydrometer is adjusted.

(ii) The corrections are expressed as units in the fourth place of decimals. The correction is to be added to or subtracted from the observed reading, according as the surface tension of the liquid under test is higher or lower than that for which the hydrometer is adjusted.

(iii) The corrections have been rounded off to the nearest 0.0001 for Series A, to the nearest 0.0002 for Series B and to the nearest 0.0005 for Series C and Series D hydrometers.

(iv) The corrections are applicable only to hydrometers conforming to Schedule No. H2, page 337, and are not applicable to specific gravity hydrometers in general.

Reading observed on the hydrometer in the sample	0.8670
Temperature of sample	17.6°C
The specific gravity $S_{17.6^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the toluole is obtained as follows:—		
Observed hydrometer reading at 17.6°C	..	0.867 0
Correction for scale error of the hydrometer	..	+0.000 4
Correction for change in volume of hydrometer from Table 15	—0.000 1
Surface tension correction from Table 16	..	0.000 0
Specific gravity $S_{17.6^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the toluole	..	0.867 3

Note that if a value accurate within ± 0.001 be adequate, all corrections, including those for scale errors of the hydrometer, could be neglected and the observed reading accepted as the specific gravity $S_{17.6^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the sample. If a value accurate to within ± 0.0005 be adequate, the temperature and surface tension corrections could safely be neglected, but it would be advisable to apply corrections for scale errors if these amounted to the maximum permissible (± 0.0004) as they would then form a large fraction of the error permissible in the final result.

The value of β , i.e. the change in density in g/ml per Centigrade degree change in temperature, for toluole is 0.00090. Hence, since

$$S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}} = S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}} + \beta(t - 15.5)$$

the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the sample of toluole is

$$\begin{aligned} &0.867\ 3 + 0.00090 (17.6 - 15.5) \\ &= 0.867\ 3 + 0.0019 \\ &= 0.869\ 2 \end{aligned}$$

II. SPECIFIC GRAVITY OF SOLIDS

Pitch is the only solid tar product for which a test for specific gravity is prescribed. The sample on which the test will be performed will consist of material which will pass a $\frac{1}{4}$ inch mesh sieve as required by the specified procedure for the sampling of pitch (see page 26). The method given below has been formulated accordingly.

Apparatus:

A standard density bottle having a nominal capacity of 100 ml (Schedule No. V11, page 365). The internal diameter

of the neck shall not be less than 12.5 mm, the figure recommended in the specification. Alternatively, a specific gravity bottle of similar capacity and into which pieces of pitch which pass a $\frac{1}{4}$ inch mesh sieve can be easily inserted, may be used.

Method:

The weight of the bottle shall be determined and a representative portion (not less than 50 g) of the sample shall be introduced. The weight of the bottle *plus* sample shall be ascertained. Freshly boiled and cooled distilled water shall be used to fill up the bottle at the specified temperature, precautions being taken that any entangled air bubbles are removed before the final adjustment, and the weight of the bottle *plus* pitch *plus* water shall be ascertained. The weight of distilled water required to fill the bottle completely at the specified temperature in the absence of pitch shall also be ascertained.

The specific gravity of the pitch shall be obtained from the resulting data, the calculation and corrections being as given in Note (i), pages 53 to 54.

SERIAL No.: G.P. 3-38

DISTILLATION PRINCIPLES

I. Definitions

The **drip-point** is the temperature at which the first drop of distillate falls from the end of the condenser. In the opinion of the Committee, the temperature reading when 5 *per cent* of the sample has distilled will indicate the presence of low boiling constituents more closely than will the drip-point, the use of which cannot be recommended.

The distillate obtained at a **stop-point** is that which is obtained when the specified temperature has been attained and, the flame having been removed or extinguished, the apparatus has been allowed to drain for (a) two minutes if the distillate coming over at the specified temperature consists essentially of low boiling products or of bases, and (b) five minutes in all other cases.

The distillate obtained at a **running-point** is that which is in the receiver when the specified temperature has been attained without interrupting the distillation at that temperature.

A specified temperature is to be regarded as having been attained immediately the top of the mercury column passes the corresponding point on the thermometer scale; this point will be the specified temperature reading or the corrected reading according to the instructions in the particular test which is being applied (see Part II below for the recommended procedure for the determination of running-points).

The **dry-point** is the temperature at which the liquid just disappears from the bottom of the flask. The flame must be removed or extinguished immediately the distillation is complete, otherwise too high a temperature will be read. The temperature reading corresponding to a distillate of 95 *per cent* of the sample is affected by small quantities of high boiling constituents and serves to detect these better than the dry-point which, under the conditions of distillation tests, is affected by superheating of the vapours, and the use of which cannot be recommended in the circumstances.

The **pitching-point** is the temperature at which (a) the temperature ceases to rise and begins to fall, and/or (b) fumes are evolved from the surface of the residue in the flask.

The **residue** in a fractionation test shall include the material which drains from the column into the flask after the removal or extinction of the flame. The draining period shall be (a) two minutes if low boiling products or bases are coming over when the flame is removed or extinguished, and (b) five minutes in all other cases.

II. Determination of Running-points

The following practice for the determination of running-points is recommended as giving accurate results while requiring the services of only one operator:—

Accurate readings of temperatures and of the corresponding volumes of distillates shall be taken at approximately regular intervals throughout the distillation and shall include readings at temperatures approximating to the specified running-points. A temperature-volume graph is constructed, from which are read the volumes of distillate at the required temperatures, the latter having been corrected for barometric pressure if the instructions so require.

III. Thermometer Readings

Corrections of thermometer readings shall be made as indicated under A, B and C below.

A. ERRORS OF THE THERMOMETER

In all cases of thermometer readings, the corrections as indicated on the certificate of the instrument shall first be made.

B. CORRECTIONS FOR BAROMETRIC PRESSURE

1. *Correction of the barometric reading:* For accurate work, the atmospheric pressure expressed in millimetres of mercury at 0°C and under standard gravity ($g = 980.665 \text{ cm/sec}^2$) requires to be determined. When these tests are carried out

TABLE 17: CORRECTION OF BAROMETER READINGS TO 0°C
FORTIN BAROMETER WITH BRASS SCALE
(The correction is to be subtracted from the barometric reading)

Temperature of barometer °C	Barometer reading (in millimetres)						
	720	730	740	750	760	770	780
	mm	mm	mm	mm	mm	mm	mm
10	1.17	1.19	1.21	1.22	1.24	1.26	1.27
11	1.29	1.31	1.33	1.35	1.36	1.38	1.40
12	1.41	1.43	1.45	1.47	1.49	1.51	1.53
13	1.53	1.55	1.57	1.59	1.61	1.63	1.65
14	1.64	1.67	1.69	1.71	1.73	1.76	1.78
15	1.76	1.78	1.81	1.83	1.86	1.88	1.91
16	1.88	1.90	1.93	1.96	1.98	2.01	2.03
17	1.99	2.02	2.05	2.08	2.10	2.13	2.16
18	2.11	2.14	2.17	2.20	2.23	2.26	2.29
19	2.23	2.26	2.29	2.32	2.35	2.38	2.41
20	2.34	2.38	2.41	2.44	2.47	2.51	2.54
21	2.46	2.50	2.53	2.56	2.60	2.63	2.67
22	2.58	2.61	2.65	2.69	2.72	2.76	2.79
23	2.69	2.73	2.77	2.81	2.84	2.88	2.92
24	2.81	2.85	2.89	2.93	2.97	3.01	3.05
25	2.93	2.97	3.01	3.05	3.09	3.13	3.17
26	3.04	3.09	3.13	3.17	3.21	3.26	3.30
27	3.16	3.20	3.25	3.29	3.34	3.38	3.42
28	3.28	3.32	3.37	3.41	3.46	3.51	3.55
29	3.39	3.44	3.49	3.54	3.58	3.63	3.68
30	3.51	3.56	3.61	3.66	3.71	3.75	3.80

in the latitude range of Great Britain, correction to standard gravity need not be made.

The atmospheric pressure shall be obtained from a reading taken on a mercury barometer having a millimetre scale, and provided with a certificate giving the index corrections of the instrument. The procedure to be adopted to correct the observed barometric reading is as follows:—

(a) *Index correction*: The observed barometer reading shall be corrected for index error in accordance with the certificate issued with the barometer. The reading thus corrected gives the atmospheric pressure as indicated by a correctly adjusted barometer at the temperature of observation and under the gravity at the place of observation and must be further corrected to give the equivalent value in millimetres of mercury at 0°C.

(b) *Reduction to 0°C*: If the mercury barometer be of the Fortin type, or of some other type in which the mercury in the cistern is set to a fiducial level when the barometer reading is taken, the temperature correction to the reading will be found in Table 17, opposite.

If the barometer used be of the Kew type, i.e. that in which the barometer reading is taken without adjusting the level of the mercury in the cistern, the temperature correction will differ somewhat from that given in Table 17. The temperature correction for a Kew pattern barometer depends to a small extent on its dimensions, but, in general, sufficient accuracy will be obtained if the correction be taken to be 5 per cent in excess of that given in Table 17 for a Fortin barometer.

(c) *Reduction to standard gravity*: The reading as corrected in accordance with the preceding paragraphs gives the pressure in terms of millimetres of mercury at 0°C at the place of observation. To obtain the equivalent pressure at 0°C and under standard gravity, the value obtained as above must be multiplied by $g/980.665$ where g is the value of gravity in cm/sec^2 at the place of observation. Changes in gravity due to causes other than change in latitude, e.g. height above sea-level, may be neglected, and the reduction to standard gravity carried out by means of Table 18, page 66, which shows appropriate corrections for different latitudes. As stated above, however, this reduction is not necessary for tests carried out in the latitudes covered by Great Britain, but Table 18 is included for use in latitudes differing more widely from the normal of 45°.

TABLE 18: CORRECTION OF BAROMETER READINGS TO STANDARD GRAVITY

Latitude	Barometer reading (in millimetres)			
	720	740	760	780
	mm	mm	mm	mm
0°	-1.93	-1.98	-2.04	-2.09
2	-1.92	-1.97	-2.03	-2.08
4	-1.91	-1.96	-2.01	-2.07
6	-1.89	-1.94	-1.99	-2.04
8	-1.85	-1.91	-1.96	-2.01
10	-1.81	-1.86	-1.92	-1.97
12	-1.76	-1.81	-1.86	-1.91
14	-1.71	-1.75	-1.80	-1.85
16	-1.64	-1.69	-1.73	-1.78
18	-1.57	-1.61	-1.65	-1.70
20	-1.49	-1.53	-1.57	-1.61
22	-1.40	-1.44	-1.48	-1.51
24	-1.30	-1.34	-1.37	-1.41
26	-1.20	-1.23	-1.27	-1.30
28	-1.09	-1.12	-1.15	-1.18
30	-0.98	-1.01	-1.04	-1.06
32	-0.86	-0.89	-0.91	-0.94
34	-0.74	-0.76	-0.79	-0.81
36	-0.62	-0.64	-0.65	-0.67
38	-0.49	-0.51	-0.52	-0.53
40	-0.36	-0.37	-0.38	-0.39
42	-0.23	-0.24	-0.24	-0.25
44	-0.10	-0.10	-0.10	-0.11
46	+0.03	+0.03	+0.04	+0.04
48	+0.17	+0.17	+0.18	+0.18
50	+0.30	+0.31	+0.31	+0.32
52	+0.43	+0.44	+0.45	+0.46
54	+0.56	+0.57	+0.59	+0.60
56	+0.68	+0.70	+0.72	+0.74
58	+0.80	+0.82	+0.85	+0.87
60	+0.92	+0.94	+0.97	+1.00
62	+1.02	+1.05	+1.08	+1.11
64	+1.14	+1.17	+1.20	+1.23
66	+1.24	+1.28	+1.31	+1.35
68	+1.34	+1.37	+1.41	+1.45
70°	+1.43	+1.47	+1.51	+1.55

NOTE: When positive (+) the correction is to be added to and when negative (-) subtracted from the barometer reading.

The following towns have approximately the latitude given against each:—

Southampton, 51°; London, 51.5°; Cambridge, 52°; Derby, 53°; York, 54°; Newcastle, 55°; Glasgow, 56°; Aberdeen, 57°.

2. *Application of the corrected barometer reading:* In all exact determinations, the observed temperature shall be corrected for the difference between the atmospheric pressure at the time of the determination and normal atmospheric pressure, this being defined as the pressure of 760 mm of mercury at 0°C and under standard gravity ($g = 980.665 \text{ cm/sec}^2$).

In the determination of boiling-points, the following relationships:—

$$C = 0.00011 (760 - P_0) (273 + t_1)$$

(which is applicable to products consisting mainly of phenolic bodies), and

$$C = 0.00012 (760 - P_0) (273 + t_1)$$

(which is applicable to products consisting mainly of hydrocarbons) give the correction C in Centigrade degrees, to be applied to the temperature t_1 , obtained by correcting the observed thermometer reading as under A, page 64, where P_0 is the barometric pressure corrected to 0°C (and standard gravity, if so corrected) obtained from (a) above. Tables 19 and 20, page 68, give the values of

$$0.00011(760 - P_0) (273 + t_1) \text{ and } 0.00012(760 - P_0)(273 + t_1)$$

respectively over a range of temperature from 0° to 425°C, and of pressure from 720 mm to 780 mm. The correction shall be added if P_0 be lower than 760 mm and subtracted if P_0 be higher than 760 mm.

C. EMERGENT STEM OF THE THERMOMETER

The standard thermometers with one exception have been specified as graduated for 100 mm immersion, measured from the bottom of the bulb, and it is understood that in use, the immersion is measured from this point to the top of the cork. For the standard flasks Schedule Nos F1 to F9, page 365, this demands that the top of the cork be *about* 10 mm above the top of the neck in order that the bottom of the capillary may be level with the lower edge of the side tube joint (see page 366).

It is not standard procedure to use thermometers calibrated for an immersion differing from that specified. When thermometers in accordance with the standard specifications are used,

STANDARD METHODS

TABLE 19: CORRECTION OF TEMPERATURE READINGS FOR BAROMETRIC PRESSURE

VALUES OF $0.00011(760 - P_0)(273 + t_1)$
(Correction to be applied for phenolic bodies)

Pressure in mm of mercury	Temperature in Centigrade degrees																	
	0	25	50	75	100	125	150	175	200	225	250	275	300	325	350	375	400	425
720	1.2	1.3	1.4	1.5	1.6	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.9	3.0	3.1
725	1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7
730	0.9	1.0	1.1	1.1	1.2	1.3	1.4	1.5	1.6	1.6	1.7	1.8	1.9	2.0	2.1	2.1	2.2	2.3
735	0.8	0.8	0.9	1.0	1.0	1.1	1.2	1.2	1.3	1.4	1.4	1.5	1.6	1.6	1.7	1.8	1.9	1.9
740	0.6	0.7	0.7	0.8	0.8	0.9	0.9	1.0	1.0	1.1	1.2	1.2	1.3	1.3	1.4	1.4	1.5	1.5
745	0.5	0.5	0.5	0.6	0.6	0.7	0.7	0.7	0.8	0.8	0.9	0.9	0.9	1.0	1.0	1.1	1.1	1.2
750	0.3	0.3	0.4	0.4	0.4	0.4	0.5	0.5	0.5	0.5	0.6	0.6	0.6	0.7	0.7	0.7	0.7	0.8
755	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.4	0.4
760	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
765	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.4	0.4
770	0.3	0.3	0.4	0.4	0.4	0.4	0.5	0.5	0.5	0.5	0.6	0.6	0.6	0.7	0.7	0.7	0.7	0.8
775	0.5	0.5	0.5	0.6	0.6	0.7	0.7	0.7	0.8	0.8	0.9	0.9	0.9	1.0	1.0	1.1	1.1	1.2
780	0.6	0.7	0.7	0.8	0.8	0.9	0.9	1.0	1.0	1.1	1.2	1.2	1.3	1.3	1.4	1.4	1.5	1.5

TABLE 20: CORRECTION OF TEMPERATURE READINGS FOR BAROMETRIC PRESSURE

VALUES OF $0.00012(760 - P_0)(273 + t_1)$
(Correction to be applied for hydrocarbons)

Pressure in mm of mercury	Temperature in Centigrade degrees																	
	0	25	50	75	100	125	150	175	200	225	250	275	300	325	350	375	400	425
720	1.3	1.4	1.6	1.7	1.8	1.9	2.0	2.2	2.3	2.4	2.5	2.6	2.8	2.9	3.0	3.1	3.2	3.4
725	1.1	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8	2.9
730	1.0	1.1	1.2	1.3	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.2	2.3	2.4	2.5
735	0.8	0.9	1.0	1.0	1.1	1.2	1.3	1.3	1.4	1.5	1.6	1.6	1.7	1.8	1.9	1.9	2.0	2.1
740	0.7	0.7	0.8	0.8	0.9	1.0	1.0	1.1	1.1	1.2	1.3	1.3	1.4	1.4	1.5	1.6	1.6	1.7
745	0.5	0.5	0.6	0.6	0.7	0.7	0.8	0.8	0.9	0.9	0.9	1.0	1.0	1.1	1.1	1.2	1.2	1.3
750	0.3	0.4	0.4	0.4	0.4	0.5	0.5	0.5	0.6	0.6	0.6	0.7	0.7	0.7	0.7	0.8	0.8	0.8
755	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.4	0.4	0.4	0.4
760	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
765	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.4	0.4	0.4	0.4
770	0.3	0.4	0.4	0.4	0.4	0.5	0.5	0.5	0.6	0.6	0.6	0.7	0.7	0.7	0.7	0.8	0.8	0.8
775	0.5	0.5	0.6	0.6	0.7	0.7	0.8	0.8	0.9	0.9	0.9	1.0	1.0	1.1	1.1	1.2	1.2	1.3
780	0.7	0.7	0.8	0.8	0.9	1.0	1.0	1.1	1.1	1.2	1.3	1.3	1.4	1.4	1.5	1.6	1.6	1.7

no correction is necessary for emergent stem. The exception is thermometer Schedule No. T3b, which is graduated for total immersion and used in Serial No. P.T. 4, page 317; in accordance with the Continental procedure in connexion with which this thermometer is used, corrections for emergent stem shall not be made.

In order to indicate that the final figure represents the corrected temperature obtained as indicated above, it is customary to record the figure thus:—

T°C (corrected)

or

T°C (corr.)

All the temperatures given in the several tests for boiling-points and distillation temperatures are to be read as having been corrected according to the above instructions, unless the contrary be stated.

2. CRUDE TAR

ANALYSIS FOR COMMERCIAL EVALUATION

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least five gallons shall be supplied for the complete analysis as described in the tests: Serial Nos. C.T. 1 to 7. If insufficient tar be available, the small scale distillation test (Serial No. C.T. 8, page 92) may be substituted for the full scale distillation test (Serial No. C.T. 7, page 77), but the results obtained thereby will not necessarily be the same as or comparable with those obtained in the full scale distillation.

Preliminary Treatment of the Sample:

In order to render samples of tar sufficiently fluid for pouring or mixing, the container may be immersed in a water-bath, the temperature of which must not exceed 60°C. The immersion shall be for the minimum period of time to secure the necessary fluidity. No source of intense heat shall be used to warm the sample.

The sample shall be thoroughly mixed immediately before any portion is withdrawn for testing, transferring it if necessary to a vessel large enough to permit adequate agitation.

If the sample should contain clear aqueous liquor in such quantity that it cannot be satisfactorily incorporated with the tar to give a uniform mixture, the weight of the combined sample and container as received shall first be determined accurately. As much as possible of the separated aqueous liquor present on the top of the tar shall be decanted and its weight and specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, shall be determined.

The remainder of the sample, in a vessel large enough to permit vigorous agitation, shall be thoroughly mixed as described above before any is withdrawn for testing. The weight of the original container shall be determined and, by difference, the weight of the sample as received. The decanted liquor shall be measured and an allowance made in any subsequent calculations of results on the basis of the "sample as received."

Specific Gravity and Density of the Tar as Received

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 and Note i on page 53 (specific gravity) or Serial No. G.P. 1, page 31 and Note ii on page 37 (density)).

In the case of samples containing excessive proportions of aqueous liquor, the test shall be performed on the thoroughly mixed tar separated therefrom as described in the last paragraph under "Preliminary Treatment of the Sample," page 71.

Specific Gravity and Density of the Anhydrous Tar

Method:

The specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, of the anhydrous tar shall be calculated from the relationship—

$$S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}} = (100 S_1 - S_2 P) / (100 - P)$$

where

S_1 = the specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, of the sample (separated from decantable liquor when present) as determined by the method described in Serial No. C.T. 1, above;

S_2 = the specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, of the decanted liquor (assumed to be also the specific gravity of the remaining liquor); and

P = the percentage by volume of the remaining liquor in the sample as determined by the method described in Serial No. C.T. 3, opposite.

If the specific gravity of any separated liquor cannot be determined, or if there be no separated liquor, the value of S_2 shall be taken as 1.01.

The density ρ_{20} of the anhydrous tar may be calculated in a similar manner, S_1 and S_2 representing in such circumstances the density, in grams per millilitre at 20°C , of the sample (separated from decantable liquor if present) and of the decanted liquor respectively.

* SERIAL No. : C.T. 3-38

Water

Apparatus:

A 100 ml graduated receiver and, in the case of tars with a water content exceeding 10 *per cent*, a further graduated receiver (e.g. Schedule No. V1 or V2, pages 341 to 343).

A glass or metal flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6, page 349).

Method:

One hundred millilitres (see Note) of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) shall be measured at laboratory temperature in the graduated receiver and transferred to the flask; the receiver shall be washed with successive quantities, in all 100 ml, of solvent naphtha (B.S. 479—1938, Part B) which shall be added to the sample in the flask.

In the case of thick tars, it is preferable to weigh accurately, directly into the flask, the quantity equal to 100 ml, adding the naphtha directly to the flask also.

The flask shall be attached to the Dean and Stark condensing and collecting system. Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray tube (see page 351). Finally, the number of millilitres of water in the graduated tube shall be noted at laboratory temperature and reported as the percentage by volume of water in the sample or in the separated tar as the case may be.

If the quantity of water should exceed the calibrated capacity of the graduated tube, small quantities must be drawn off from time to time and the whole of the water finally collected in a suitable graduated receiver (e.g. Schedule No. V1 or V2, pages 341 to 343) where it shall ultimately be measured at laboratory temperature.

Note:

If the percentage of water by weight be required, 100 g of the thoroughly mixed sample or of the thoroughly mixed separated tar shall be used for the test. Assuming the density 1.00 g/ml for the water collected in the graduated tube, the number of millilitres of water collected at laboratory temperature shall be reported as the percentage by weight of water in the sample or in the separated tar as the case may be.

SERIAL NO.: C.T. 4-38

Insoluble Matter ("Free Carbon")**Method:**

Two grams of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) shall be warmed on the water bath and mixed intimately in a 200 ml beaker with 100 ml of hot pure toluole (B.S. 805—1938, Part A) (see Notes i and ii). After settlement for twenty minutes with the beaker standing on the water bath, the supernatant liquid shall be decanted cautiously, avoiding disturbance of the sediment, either (a) through balanced filter papers (e.g. Whatman No. 5) about 150 mm in diameter, or (b) through a weighed Gooch crucible prepared with asbestos and used under vacuum.

If filter papers are used, they must be heated to a temperature between 95° and 110°C and reduced to equal weight by removing the apex of the heavier paper; the uncut paper is used inside the originally heavier paper. The separated material is retained by the inner paper, but both of them are equally subjected to any action exerted by the toluole.

Washing shall be effected repeatedly by *decantation* until 300 ml of the toluole have been used. The residue shall be transferred to the filter and the washing continued until altogether 500 ml have been used. The residue shall be dried in an oven at a temperature between 95° and 110°C until it is of constant weight. The residue shall be incinerated, the weight of the ash deducted (after allowing for that in the filter paper) and the percentage by weight of ash-free "free carbon" in the anhydrous tar shall be calculated and reported.

Notes:

(i) The temperature of the toluole throughout the determination must be between 90° and 100°C.

(ii) *If the above method be varied by the use of any solvent other than hot pure toluole to the above mentioned specification, the result shall be reported as "Matter insoluble in —" and not as "free carbon."*

SERIAL No.: C.T. 5-38

Chlorine (Calculated as Ammonium Chloride)

General:

The tar is completely burned in the presence of Eschka mixture; the chlorine is then determined volumetrically.

Apparatus:

A platinum crucible of about 30 ml capacity.

A muffle furnace of appropriate size.

Reagents, etc.:

Eschka mixture, prepared by intimately mixing two parts by weight of magnesium oxide with one part of anhydrous sodium carbonate.

Standardized 0.05N solutions of silver nitrate and potassium thiocyanate.

Indicator solution prepared from a cold saturated aqueous solution of ferric ammonium alum by the addition of sufficient nitric acid to cause the disappearance of the brown colour.

Method:

About 8 g of the Eschka mixture shall be placed in the crucible and the total weight determined; about 3 g of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) shall be added and the total weight determined to give, by difference, the weight of tar used. The contents of the crucible shall be intimately mixed by means of a small glass rod which shall be finally scraped to remove any adhering material. The contents of the crucible shall be covered with about 2 g of the Eschka mixture.

The crucible and its contents shall be heated in the muffle furnace, the heat being applied gently at first; after about one hour the crucible shall be raised to a dull red heat and shall be so maintained for two hours. The crucible shall be allowed to cool and the contents transferred to a beaker, using about 50 ml of chlorine-free distilled water for the purpose. The beaker shall be covered with a clock glass and 35 ml of pure

nitric acid cautiously added. The solution shall be warmed and a measured excess of the 0.05N silver nitrate solution added, after which the whole shall be put aside to settle in a dark cupboard, preferably overnight.

After settlement, the contents of the beaker shall be filtered and the filter paper washed with five consecutive quantities of 20 ml each of chlorine-free distilled water applied from a fine jet. The excess of silver nitrate in the filtrate shall be determined by titration with the 0.05N potassium thiocyanate solution using 2 ml of the iron alum solution as indicator. The amount of silver nitrate solution used in the precipitation of silver chloride shall be determined by difference.

A blank determination shall be carried out on the reagents. This determination is preferably carried out at the same time as the main analysis.

The percentage by weight of chlorine, expressed as ammonium chloride in the sample or in the separated tar as the case may be, is—

$$\frac{(V - V_0) \times 0.002875 \times 100}{W}$$

where

V = the number of millilitres of 0.05N silver nitrate solution used in the test on the sample;

V₀ = the corresponding number of millilitres of 0.05N silver nitrate solution used in the blank determination; and

W = the weight in grams of sample or separated tar used.

SERIAL NO.: C.T. 6-38

Ash

Apparatus:

A standard distillation flask (Schedule No. F4, page 365).

A standard air condenser (Schedule No. C3, page 370).

A 100 ml graduated receiver (e.g. Schedule No. V1, page 341).

A platinum or porcelain crucible; the crucible used for the determination of the volatile matter in pitch (Serial No. P.T. 6, page 326) is suitable.

Method:

One hundred grams of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) shall be weighed into the weighed distillation flask and 50 ml of dry heavy naphtha shall

be added. The condenser shall be connected to the flask, and the contents of the latter shall be distilled over a naked flame until 100 ml of distillate have collected, when the distillation shall be stopped. The weight of the flask and residual pitch and, by difference, the weight of the pitch shall be determined.

The ash content of the residual pitch shall be determined by incinerating one gram to constant weight in the platinum or porcelain crucible. The percentage ash content of the anhydrous tar shall be obtained by calculation.

SERIAL No.: C.T. 7-38

Distillation (Full Scale)

General:

The complexity of and variations between crude tars will make it difficult to adhere in all cases strictly to any *detailed* scheme of analysis following the primary distillation. The composition and yields of the fractions from tars of various origins may differ considerably.

The scheme described below is intended to form a basis for the analysis of crude tars. A primary distillation is followed by a detailed examination of the several fractions obtained, including extraction of the phenols and fractionation of the resulting washed oil.

The three charts on pages 78 and 79 illustrate the scheme and correspond to three broad groups of the analyst's requirements, viz. :—

Chart I represents the **primary distillation**.

Chart II covers additionally the **extraction of crude phenols, naphthalene and anthracene**, together with a fractionation for evaluation purposes and for checking yields against large scale operations.

Chart III illustrates the **more complete analysis**, giving yields of benzole, toluole and naphthas, as well as pyridine bases and phenolic fractions in approximately commercial grades. This scheme of analysis should be useful in providing a standard procedure for the detailed analysis of crude tars.

If a detailed analysis of the separate products (such as benzole, phenols or bases) be required, it may be necessary to distil a considerably larger volume of crude tar than the 5 litres specified under "Method" below, in order to provide sufficient

STANDARD METHODS

CHART I: PRIMARY DISTILLATION OF CRUDE TAR

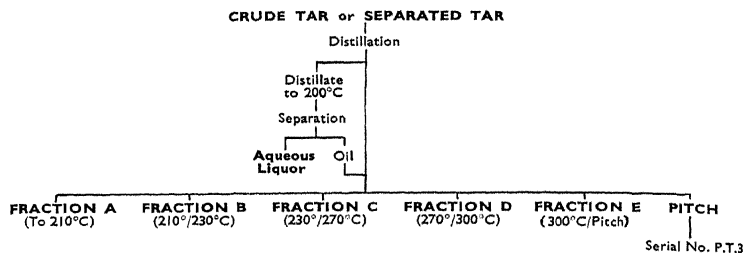
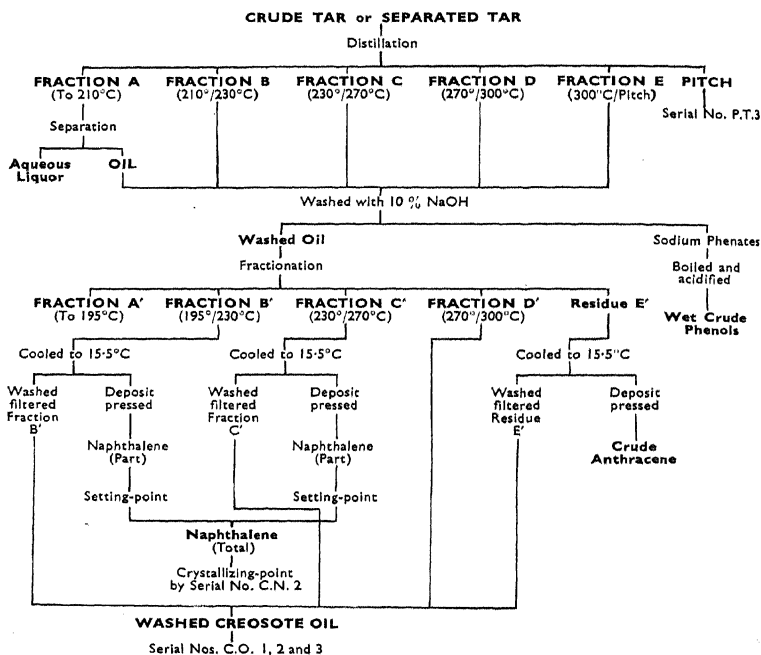


CHART II: PARTIAL ANALYSIS OF CRUDE TAR

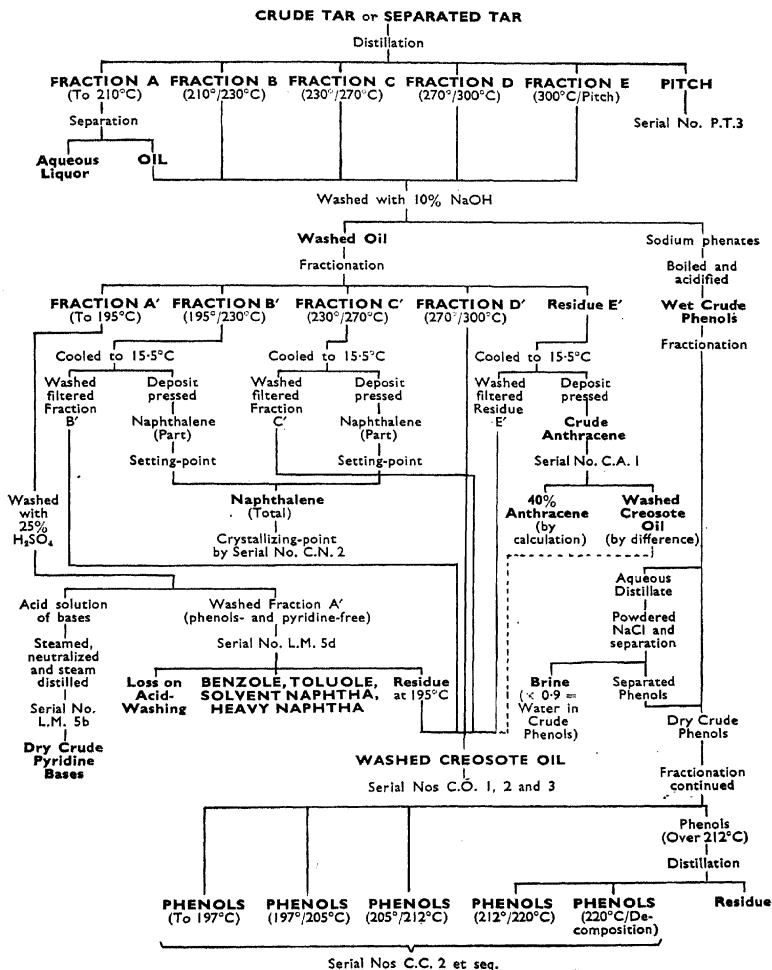


NOTE.—Products shown in heavy type full capital letters (e.g. **CRUDE TAR**) are weighed and their specific gravities are determined. Products shown in heavy type initial capital letters (e.g. **Aqueous Liquor**) are weighed but their specific gravities are not determined.

CRUDE TAR

79

CHART III: FULL ANALYSIS OF CRUDE TAR



NOTE.—Products shewn in heavy type full capital letters (e.g. **CRUDE TAR**) are weighed and their specific gravities are determined. Products shewn in heavy type initial capital letters (e.g. **Aqueous Liquor**) are weighed or their quantities otherwise determined but their specific gravities are not determined.

distillate for isolation of the required product or products. In such circumstances, the corresponding fractions from successive charges shall be thoroughly mixed before further examination.

The laboratory scale distillation of crude tar inevitably gives rise to some degree of cracking; the yields of the several fractions and the results of the subsequent examination of such fractions may consequently be inconvertible directly to large scale

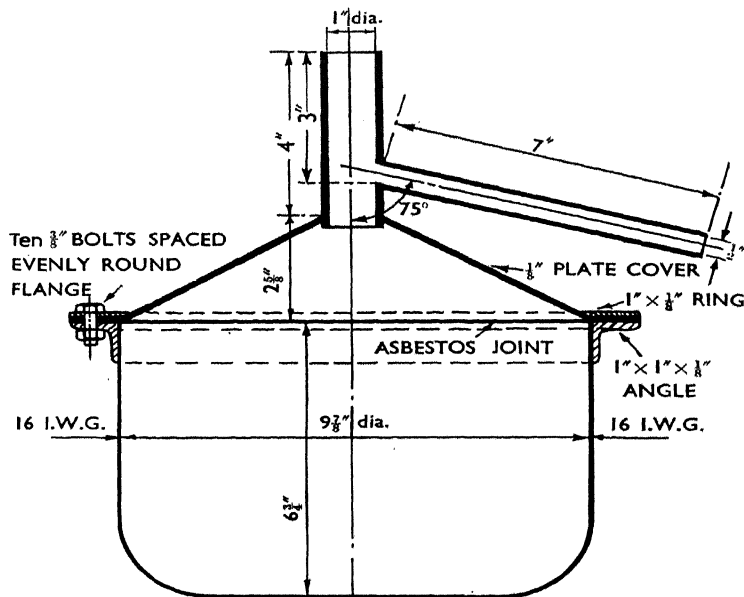


Fig. 9.—Full Scale Distillation of Crude Tar—Tar Still.

practice. In any event, no one laboratory analysis could be devised to give such comparability, because of the variations in works' practice.

Apparatus:

A still and support as shewn in Figs 9 above and 10 opposite. The upper part of the still shall be covered with a hood of asbestos, about $\frac{1}{8}$ inch thick, as shewn in Fig. 11, page 82.

An asbestos lined screen, constructed in two halves which may be hinged, arranged to surround the still as shewn in Fig. 11.

An air condenser, consisting of a straight glass tube, $750 \text{ mm} \pm 10$ in length and $25 \text{ mm} \pm 2$ in internal diameter, fitted at one end with the cone of a standard B24 ground glass joint and further ground at the same end at an angle of about 45° to the axis of the tube.

A Liebig condenser $600 \text{ mm} \pm 10$ in length and $14 \text{ mm} \pm 1$ in internal diameter, and having a water jacket $450 \text{ mm} \pm 10$ in length. The open end of the wide portion of the inner tube

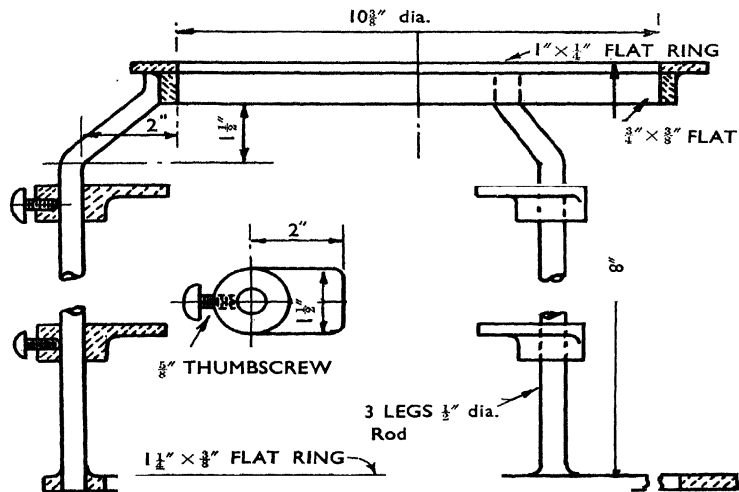


Fig. 10.—Full Scale Distillation of Crude Tar—Support for Tar Still.

shall be fitted with the socket of a standard B24 ground glass joint. The junction between the wide and narrow portions of the inner tube must be drawn out in such a way as to eliminate any sharp angles where distillate may lodge.

The Liebig condenser shall be connected to the delivery end of the air condenser by means of the ground glass joint.

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333), so fitted in the still that the bottom of the capillary is level with the lower edge of the side tube joint.

Receivers consisting of weighed vessels of suitable capacity.

The still is heated by two burners: (a) a ring burner surrounding the still; and (b) a Fletcher standard No. 30 double ring burner underneath the still.

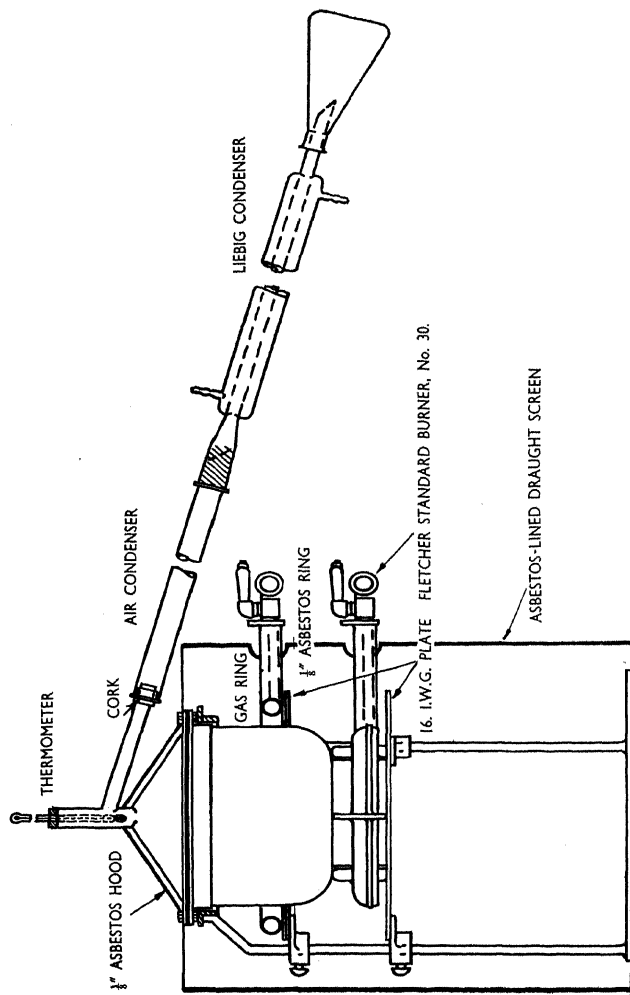


Fig. 11.—Full Scale Distillation of Crude Tar—Assembled Apparatus.

Method:**DISTILLATION**

A weight of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) corresponding to 5 litres at 20°C shall be charged into the weighed still.

The distillation shall be carried out slowly while water is distilling, the heating being carried out principally by means of the upper ring burner placed so that the flame apertures are about 100 mm below the flange of the still. Only a small flame shall be maintained on the bottom of the still during this period (see Note).

When oil is distilling freely, the upper flame shall be extinguished and the heating by the lower burner increased so that distillation is as rapid as is compatible with complete condensation of the vapours; the rate must not be allowed to fall below 9 ml per minute during this stage. The distillates shall be collected in weighed vessels of suitable capacity.

No correction need be made for barometric pressure in this distillation and fractions shall be taken as follows:—

Crude fraction A	Up to 210°C
Crude fraction B	210° to 230°C
Crude fraction C	230° to 270°C
Crude fraction D	270° to 300°C
Crude fraction E	300°C to pitch (see under "Pitch," page 90)

all the temperatures being running-points except the pitching-point, which shall be a stop-point.

The Liebig condenser shall be removed without interrupting the distillation when the thermometer records 190°C.

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

The fraction up to 210°C, warmed if necessary, shall be transferred to a separator and after settling, the liquor and the oil shall be separated, adding powdered sodium chloride if necessary to obtain a clear separation of the two layers. The weight and specific gravity of the oil layer shall be determined. The weights and specific gravities of the remaining liquid fractions and of the pitch remaining in the still shall be determined.

Note:

If the above primary distillation be alone carried out, a greater accuracy will be obtained by stopping the distillation at 200°C, allowing the distillate to settle, separating the oil from the aqueous distillate and returning the oil to the still when the latter has cooled sufficiently and before restarting the distillation.

The methods detailed in the following paragraphs for the examination of the fractions obtained by the foregoing distillation are recommended when a full analysis of the crude tar is required, but at this stage the operator is advised to consider the detailed procedure which shall be adopted having regard to any special information which may be required.

PHENOLS

The distillates (after separating the aqueous liquor in the case of crude fraction A as described above) shall be thoroughly mixed together. It may be necessary to warm the distillates in order to bring into solution any solid which has crystallized out. The volume of the mixed distillates shall be noted; the mixture shall be transferred to a stoppered separating funnel of suitable size and the phenols extracted as follows:—

An equal volume of a 10 *per cent w/w* clear aqueous caustic soda solution shall be used to rinse the vessels from which the separate and mixed distillates were transferred, and then added to the latter in the separating funnel. If the mixture should contain solid hydrocarbons which tend to separate, both the mixture and the caustic soda solution shall be warmed just sufficiently to maintain the hydrocarbons in solution. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement, the alkaline layer shall be run into a beaker. The upper layer shall be agitated for five minutes with a further quantity of the caustic soda solution, equal to one quarter of the volume of the mixture which is being washed, and with further successive quantities of the caustic soda solution, each equal in volume to the last, until all the phenols have been removed. This can be ascertained by *slightly* acidifying the last washings, using concentrated hydrochloric acid, and examining for separated phenols. After settlement, the alkaline layer shall in each case be added to the first soda washing. A large excess of soda must be avoided but some

excess must be maintained; as a rough guide it may be assumed that 25 ml of caustic soda solution of the strength indicated are sufficient to remove about 5 ml of phenols. During the washing processes, the contents of the funnel shall be kept liquid, immersing the funnel in warm water if necessary.

The soda washings, including any acidified for the purpose of checking the washing procedure, shall be combined. The combined washings shall be alkaline; they shall be boiled vigorously for twenty minutes; fragments of porous or other suitable inert material may be used to prevent bumping. The soda washings shall be cooled to laboratory temperature and filtered through glass wool previously moistened with saturated aqueous sodium chloride solution; the glass wool shall be washed with 25 ml of saturated sodium chloride solution which shall be added to the filtered soda washings. Methyl orange indicator solution shall be added, followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the mixture shall be kept cool by immersing the container from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a *few* particles undissolved. The whole shall be transferred to a suitable separator and after adequate time for settlement the phenols shall be separated; their volume shall be reported as the content of "wet crude phenols" in the distillate.

The apparatus described for the initial stage of the method Serial No. C.C.4, page 208, shall be assembled as indicated therein, except that the round bottomed flask shall be of suitable size for the distillation of the combined wet crude phenols. These phenols shall be placed in the flask and distilled slowly until all the water has come over (i.e. until the drops at the end of the condenser cease to be turbid). The phenols in the distillate shall be salted out by the addition of powdered sodium chloride in just sufficient quantity to saturate the aqueous layer and leave a *few* particles undissolved. The aqueous layer shall be measured and the phenols separated; the volume of the aqueous layer, multiplied by 0.9, shall be taken as the water content of the phenols (see Note).

The contents of the flask shall be allowed to cool to about 100°C and the phenols separated from the distillate shall be returned to the flask. The distillation shall be continued at the

rate of 2 to 2.5 ml per minute (about one drop per second) and fractions taken separately as follows:—

Up to 197°C
197° to 205°C
205° to 212°C

197°C and 205°C being running-points and 212°C being a stop-point.

The column shall be removed and the flask shall be fitted with a cork carrying a suitable thermometer (e.g. Schedule No. T4a, page 333) and glass bend connecting the flask directly to the air condenser. The thermometer shall be so fitted that the immersion mark is level with the top of the cork. The exit tube shall extend 10 mm beyond the bottom of the cork in the flask and at least 25 mm beyond the cork in the upper end of the condenser. The distillation shall be resumed and further fractions shall be taken as follows:—

212° to 220°C
220°C to pitching-point

220°C being a running-point and the pitching-point (stop-point) being recorded.

All the fractions shall be weighed and their specific gravities determined; any further examination of these crude fractions shall be carried out by the appropriate methods given in Serial Nos C.C. 2 *et seq.* (pages 203 *et seq.*).

Note:

If the water content of the wet crude phenols be alone required and the fractionation of the dry crude phenols is to be omitted, a portion of the wet phenols may be examined according to the method described in Serial No. C.C. 7, page 216.

FRACTIONATION OF WASHED OIL

The warmed washed oil, or an aliquot portion thereof, shall be transferred to a weighed bolt head flask of suitable size; a standard 12-bulb pear column (Schedule No. A2, page 370) shall be fitted to the flask and a standard Liebig condenser with bent end (Schedule No. C1, page 369) shall be fitted to the side arm of the column. The flask shall be supported at the neck by means of a clamp. The column shall not be lagged in any way.

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333) shall be so fitted in the top of the

column that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S2, page 373) shall be placed round the flask and the assembled apparatus adjusted so that the column is vertical.

The contents of the flask shall be distilled at the rate of 2 to 2.5 ml per minute (about one drop per second). The following fractions shall be collected in weighed receivers of suitable size:—

Fraction A'	..	Up to 195°C (stop-point)
Fraction B'	..	195° to 230°C (running-point)
Fraction C'	..	230° to 270°C (running-point)
Fraction D'	..	270° to 300°C (stop-point)

When the temperature reaches 195°C, the gas supply shall be reduced in order to stop the distillation temporarily and the Liebig condenser shall be replaced by the standard air condenser (Schedule No. C3, page 370). The gas supply shall then be increased and the distillation continued at the same rate as before (2 to 2.5 ml per minute).

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

The weights and specific gravities of the liquid fractions shall be determined. The residue at 300°C in the flask (residue E') shall be cooled and weighed and retained for separation of the crude anthracene.

PYRIDINE BASES

The distillate to 195°C (fraction A') shall be transferred to a separating funnel of suitable size and shaken for two minutes with a fifth of its volume of 25 *per cent w/w* sulphuric acid. The mixture shall be allowed to stand until separation is complete.

The lower layer shall be run into a round bottomed flask of suitable size and the distillate washed with the same volume of 25 *per cent w/w* sulphuric acid as before. The combined acid washings shall be boiled for ten minutes and after cooling, sufficient 20 *per cent w/w clear* aqueous caustic soda solution shall be added to neutralize the whole of the acid used. The mixture shall be kept cool during neutralization, to avoid loss of pyridine bases.

One hundred millilitres of water shall be added and the mixture distilled in the apparatus used in Serial No. C.C.5 (see

Fig. 23, page 213), except that the receiver shall be a flask of suitable size; distillation shall proceed until the drops of condensate shew only a faint opalescence.

The condensate shall be treated for the liberation of the pyridine bases which shall be measured if sufficient in quantity, or shall be titrated for the content of pyridine bases according to part A or part B of the method described in Serial No. L.M. 5b, page 135.

BENZOLE, TOLUOLE AND SOLVENT NAPHTHAS

The distillate to 195°C (fraction A') from which the liquor, phenols and pyridine bases have been removed, shall be washed with 95 *per cent w/w* sulphuric acid and fractionated as described in Serial No. L.M. 5d, page 138, the residue at 190°C being part of the washed creosote oil (see "Washed Creosote Oil," page 90).

CRUDE NAPHTHALENE

It is to be understood that this determination is only in respect of the naphthalene deposited from solution at 15.5°C.

The distillate from 195° to 230°C (fraction B') and, if it should contain crystalline solid on cooling, the distillate from 230° to 270°C (fraction C') shall be examined separately as follows:—

Fifty grams, or more if the naphthalene content is expected to be low, shall be cooled to $15.5^{\circ}\text{C} \pm 0.5$ Centigrade degree and after seeding with a small crystal of naphthalene if no crystalline material has appeared by then in the cooled distillate, shall be maintained within those limits of temperature for four hours, with occasional stirring.

The material shall then be transferred to a cold Buchner funnel fitted with a rapid filter paper, and as much oil as possible removed rapidly by a filter pump. The naphthalene shall be placed between sheets of blotting paper and submitted to pressure in a hand screw-press. The oily margins of the cake shall be removed and separately pressed.

The operations, starting with the transfer to the Buchner funnel to the preliminary pressing, shall be conducted with the utmost rapidity.

The pressed naphthalene shall be weighed and its "corrected wet crystallizing-point" shall be determined by the method described in Serial No. C.N. 2, page 248. The crystallizing-point shall be at least 70°C.

The amount of crude naphthalene in the original tar shall be calculated from the combined weights in the washed fractions B' and C'. The crystallizing-point to be reported shall be that of an intimate mixture of equal aliquot portions of the naphthalene from the two washed fractions.

The approximate percentage of pure naphthalene in the pressed naphthalene may, if desired, be calculated as—

$$2t - 60$$

where

t = the "corrected wet crystallizing-point" of the pressed naphthalene.

The above formula is, for the present purpose, sufficiently in agreement with that appearing in Serial No. C.N. 3, page 250. The appearance here of the relationship there shewn would give a misleading impression of the value of the results obtained in the present connexion.

CRUDE ANTHRACENE

It is to be understood that this determination is only in respect of the anthracene deposited from solution at 15.5°C.

The residue over 300°C from the fractionation, or 50 g thereof, shall be cooled to $15.5^{\circ}\text{C} \pm 0.5$ Centigrade degree and, after seeding with a small crystal of anthracene, shall be maintained within those limits of temperature for four hours, with occasional stirring.

The material shall then be transferred to a cold Buchner funnel fitted with a rapid filter paper, and as much oil as possible removed rapidly by a filter pump. The crude anthracene shall be placed between sheets of blotting paper and submitted to pressure in a hand screw-press. The oily margins of the cake shall be removed and separately pressed and the procedure repeated until no further oil is removable.

The operations, starting with the transfer to the Buchner funnel to the preliminary pressing, shall be conducted with the utmost rapidity.

The pressed anthracene shall be weighed and the amount in the sample itself calculated; its content of pure anthracene shall be determined by the method described in Serial No. C.A. 1, page 299. From the result, the equivalent weight of 40 *per cent* anthracene shall be calculated and reported, the difference between this weight and that of the pressed

anthracene being part of the *total washed* creosote oil (see below).

WASHED CREOSOTE OIL

The residue at 300°C from the fractionation after removal of crude anthracene; the residue at 190°C in the fractionation of fraction A'; fractions B' and C' after removal of naphthalene; and the distillate from 270° to 300°C (fraction D') together constitute the *available washed* creosote oil. They, or equal aliquot portions thereof, may be blended and tested by the methods described in Serial Nos C.O. 1, 2 and 3, pages 269 and 270.

Due allowance must be made for oil losses in pressing the crude naphthalene and anthracene. An approximation to the *total washed* creosote oil may be made by deducting from the total weight of the residue over 300°C and fractions B', C' and D', the total of the weights of the naphthalene recovered and the 40 *per cent* anthracene calculated as above.

PITCH

Unless otherwise required, the pitch content shall be recorded in terms of material giving a result of 70°C when examined by the Kraemer and Sarnow test, Serial No. P.T. 3, page 311. If, when this test is applied to the material in the still, the result be not 70°C, the figure determined as the weight of the pitch remaining in the still may be adjusted so that the recorded figure represents the content of pitch of the standard quality; in such circumstances, the figure for the weight of fraction E shall be adjusted equally in the opposite direction. For the purpose of calculation, it shall be assumed that the addition of fraction E to the extent of 1 *per cent* of the pitch yield lowers the Kraemer and Sarnow result by 2 Centigrade degrees. This adjustment may however only be applied if the Kraemer and Sarnow test on the residue in the still gives a figure within the range 60° to 75°C inclusive.

If the grade of the pitch be determined by one of the other standard methods and the weight of pitch requires similar adjustment, it shall be assumed that the addition of fraction E to the extent of 1 *per cent* of the pitch yield lowers the Ring and Ball result (Serial No. P.T. 4, page 317) by 2 Centigrade degrees, and the Half Inch Cube result (Serial No. P.T. 5, page 322) by 2.5 Centigrade degrees. These corrections may also only be applied when the test gives a figure not more than 10 Centigrade degrees below or 5 Centigrade degrees above the standard.

CRUDE TAR

91

STATEMENT OF RESULTS

The analytical results from the series of tests described in Serial Nos 1 to 7 shall be recorded as in Table 21, below.

TABLE 21: ANALYSIS FOR COMMERCIAL EVALUATION OF CRUDE TAR
(including Full Scale Distillation Test)

	Specific gravity S _{15.5°C/15.5°C}	Percentage on sample as received	Per ton or per 1,000 gal. of sample as received
Sample as Received		% by volume	
Crude tar as received		—	—
Separated liquor			gal.
Separated tar			gal.
Unseparated liquor	—		gal.
Anhydrous tar			gal.
Chlorine (calculated as ammonium chloride)	per cent by weight	
Analysis of the Tar		% by weight	
Total liquor	—		—
Crude fraction A: Up to 210°C			gal.
Crude fraction B: 210° to 230°C			gal.
Crude fraction C: 230° to 270°C			gal.
Crude fraction D: 270° to 300°C			gal.
Crude fraction E: 300° to °C			gal.
Pitch, K. & S. 70°C()*			cwt
Loss by difference	—		—
Refractionation of Washed Crude Distillates		% by weight	
Fraction A': Up to 195°C			gal.†
Fraction B': 195° to 230°C			gal.†
Fraction C': 230° to 270°C			gal.†
Fraction D': 270° to 300°C			gal.†
Residue: Above 300°C	—		gal.†
Partially Rectified Products		% by weight	
Loss on acid-washing	—		gal.
"Benzole" fraction: Up to 100°C			gal.
"Toluole" fraction: 100° to 120°C			gal.
Solvent naphtha: 120° to 160°C			gal.
Heavy naphtha: 160° to 190°C			gal.
Phenols: Up to 197°C			gal.
Modified Lowe's test: °F	—	—	—
Phenols: 197° to 205°C			gal.
Phenols: 205° to 212°C			gal.
Phenols: 212° to 220°C			gal.
Phenols: 220° to °C			gal.
Dry crude pyridine bases	—		gal.
Total washed creosote oil			gal.
Crude naphthalene (crystallizing-point: °C)			lb.
Crude anthracene (calculated as 40 per cent anthracene)			lb.
Ash-free "free carbon":	per cent by weight on anhydrous tar		
Ash:	per cent by weight on anhydrous tar		
<p>* Indicate here whether the figure for pitch of K. & S. 70°C is "actual" (the pitch obtained in the test being of K. & S. 70°C) or "calculated" (the pitch in the still being of K. & S. other than 70°C, the amount having been calculated to the basis of K. & S. 70°C by means of the relationship given in the test).</p> <p>† Fractions which cannot be conveniently measured by volume, by reason of their high temperature of complete fluidity, are to be returned in hundredweights in the third column.</p>			

Distillation

(Small Scale)

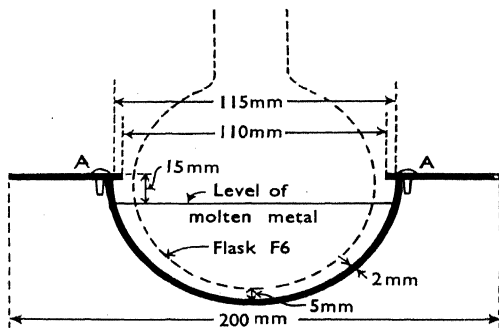
Apparatus:

A standard distillation flask (Schedule No. F6, page 365).

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333), so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard air condenser (Schedule No. C3, page 370).

A metal heating bath, as shewn in Fig. 12, below. The bath shall contain fusible alloy melting below 70°C , in such quantity that, when the bottom of the flask is 5 mm from the bottom of the bath, the level of the molten metal is about 15 mm below the rim of the bath.



Dimensions are minima.

A, A: Three or four pins or similar device to retain flange central, if flange and bowl not in one piece.

Fig. 12.—Small Scale Distillation of Crude Tar—Heating Bath.

Crow receivers (Schedule Nos V1 to V3, page 341) of appropriate size.

Method:

A known volume of the thoroughly mixed sample or of the thoroughly mixed separated tar (see under "Preliminary Treatment of the Sample," page 71) shall be distilled from a

distillation flask of suitable size, using a standard Liebig condenser (Schedule No. C2, page 369), until water ceases to come over. The water shall be separated from the accompanying oils, after adding powdered sodium chloride if necessary to obtain a clear separation of the two layers, and the oil shall be returned to the distillation residue when that residue has cooled to about 40°C; the whole shall be thoroughly mixed, and about 250 ml shall be accurately measured into the weighed distillation flask specified under "Apparatus." The bottom of the flask shall be smoked in a luminous flame and, the fusible metal having been heated to a temperature slightly higher than its melting-point, the flask shall be immersed centrally in the bath; the bottom of the flask shall be at least 5 mm above the bottom of the bath. The assembly of the apparatus shall be completed, with the thermometer and air condenser specified under "Apparatus."

The distillation shall be carried out steadily at the rate of 5 ml per minute and fractions collected separately in the Crow receivers, without interrupting the distillation, as follows:—

- Up to 210°C
- 210° to 230°C
- 230° to 270°C
- 270° to 300°C
- 300°C to pitch (see under "Pitch," below)

the final distillation temperature being recorded.

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

PITCH

The weight and specific gravity of the pitch remaining in the flask shall be determined. The grade of the pitch, as determined by one of the standard pitch tests (Serial Nos P.T. 3, 4 and 5, pages 311 to 322), shall be recorded; if it be desired to correct the quantity of pitch to that of a standard grade, the adjustment may be made as described under "Pitch" in Serial No. C.T. 7, page 90, provided the actual result of the test on the pitch is within the tolerable range there indicated. The quantity of the final distillate will in such circumstances require to be correspondingly corrected in the opposite direction.

Fraction	Specific gravity S _{15-5°C/15-5°C}	Condi- tion after twenty- four hours at 15-5°C	Caustic soda extract: <i>per cent</i> by volume	Percentage on sample as received		Content on anhydrous tar per ton or per 1,000 gal.
				by volume	by weight	
Up to 210°C						gal.
210° to 230°C						gal.
230° to 270°C						gal.
270° to 300°C						gal.
300° to °C						gal.
Pitch		(Serial No. P.T. ^{test} } : °C				cwt
Loss on distillation: <i>per cent</i> by weight						
Ash-free "free carbon": <i>per cent</i> by weight on anhydrous tar						
Ash: <i>per cent</i> by weight on anhydrous tar						

occasional stirring; each shall then be examined for solid matter, and the result of this observation shall be reported.

CAUSTIC SODA EXTRACT

Each oil fraction shall be separately treated as follows:—

Ten millilitres, or the whole fraction if less than 10 ml be available, shall be run into a 50 ml stoppered cylinder graduated to 0.2 ml; the fraction shall be warmed if necessary until completely liquid, its temperature when measured being noted. Ten millilitres of solvent naphtha (B.S. 479—1938, Part B) shall be added, followed by a volume of 10 *per cent w/w clear* aqueous caustic soda solution equal to three times the volume of oil taken. The cylinder shall be stoppered and the contents mixed thoroughly until no further reduction occurs in the volume of the oil layer. The contents of the cylinder shall be allowed to settle and the volume of residual oil read at the same temperature as that at which the oil was measured in the cylinder. The percentage loss in volume of the oil layer shall be calculated and reported as the “caustic soda extract.”

STATEMENT OF RESULTS

The analytical results shall be recorded as in Table 22, page 94.

3. REFINED TAR, INCLUDING ROAD TAR

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least a gallon (about 4,500 ml) shall be supplied for an examination by the following tests: Serial Nos R.T. 1, 2 and/or 3, 4, 5, 6, 7 and 8.

Preliminary Treatment of the Sample:

In order to render samples of tar sufficiently fluid for pouring or mixing, the container may be immersed in a water-bath, the temperature of which must not exceed 60°C. The immersion shall be for the minimum period of time to secure the necessary fluidity. No source of intense heat shall be used to warm the sample.

The sample shall be strained through a 20-mesh sieve and the presence of any solid matter on the sieve shall be reported.

The sample shall be thoroughly mixed immediately before any portion is withdrawn for testing, transferring it if necessary to a vessel large enough to permit adequate agitation.

SERIAL No.: R.T. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 and Note i on page 53 (specific gravity) or Serial No. G.P. 1, page 31 and Note ii on page 37 (density)).

SERIAL No.: R.T. 2-38

Viscosity

Apparatus:

The tar viscometer (Figs 13 and 14, pages 98 and 99) is a modified form of the Redwood viscometer and consists essentially of a cup having a standard orifice and valve, and a water-bath.

The cup is constructed of a length of hard brass tube of the precise dimensions shewn in Fig. 15, page 100. An external brass collar is fitted to the upper (open) end of the cylinder; this serves to support the cup in the sleeve of the water-bath. The bottom of the cup consists of a circular phosphor-bronze

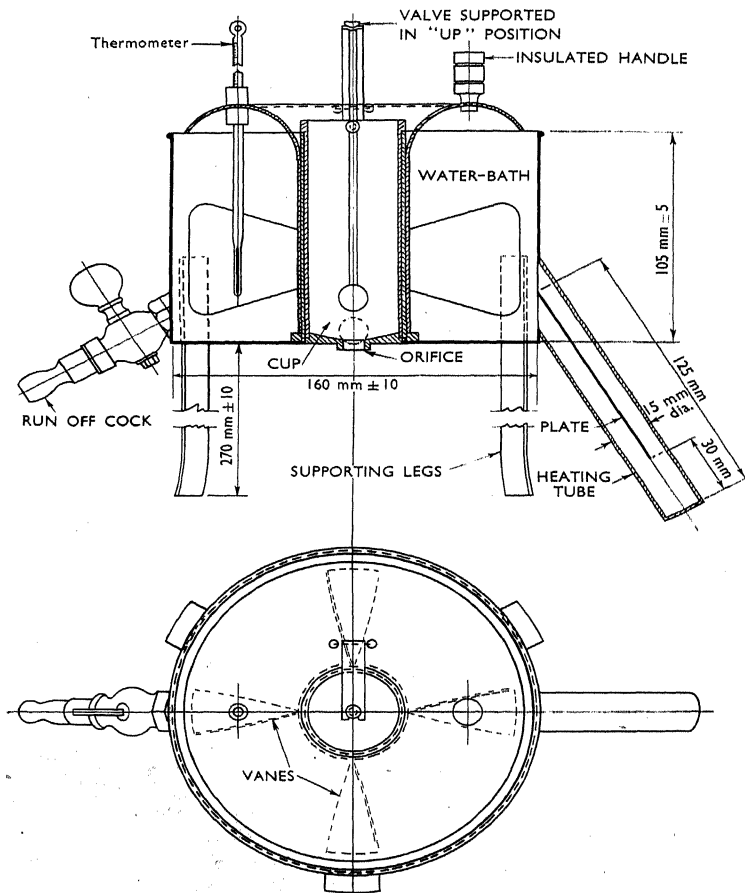


Fig. 13.—Viscosity of Refined Tar—Elevation and Plan of Assembled Viscometer.

plate screwed into the cylinder and made conical to facilitate drainage of the tar after use. It is provided centrally with a perfectly cylindrical phosphor-bronze (90/10 cast) extension which must be drilled and polished internally to provide an orifice precisely to the specified dimensions. The upper rim of the orifice must be accurately circular, in order to provide a seating for the valve. The cup shewn in Fig. 15 and having an orifice of 10 mm is subsequently referred to as the 10 mm cup.

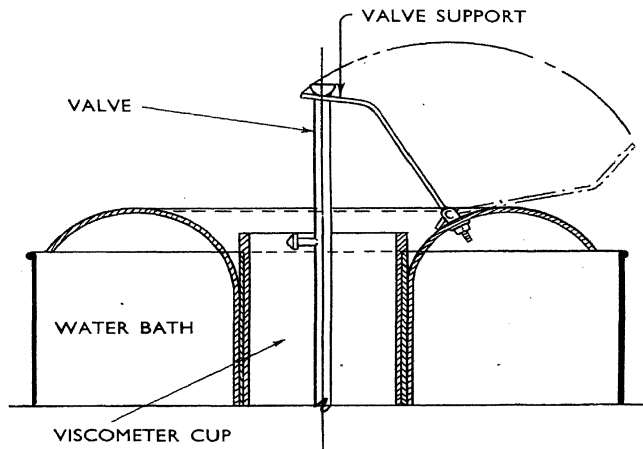


Fig. 14.—Viscosity of Refined Tar—Section shewing Arrangement of Valve Support.

The valve which serves to close the orifice of the tar cup is a phosphor-bronze sphere attached to a Monel metal rod. The rod is provided with a levelling peg and at its upper end with a hemisphere by which the valve is held in the valve support (see below). The valve shall be constructed precisely to the dimensions shewn in Fig. 15, page 100.

The water-bath is cylindrical, about 160 mm in diameter and about 105 mm in depth. It is made of copper sheet and is provided with a small brass run-off cock. There is also a side tube, about 125 mm in length and about 15 mm in diameter, to which the flame of a burner may be applied for maintaining the temperature of the water. This tube is bisected by a

longitudinal plate to about 30 mm from the closed lower end. Both the run-off cock and the side tubes are brazed or soldered to the bath as shewn in Fig. 13, page 98, and the internal joints must be smooth and free from projecting metal which might otherwise interfere with normal convection. A stout brass tube, 105 mm in height and about 42.1 mm in internal diameter, is brazed or soldered into a central hole cut in the bottom of the bath; this furnishes a sleeve to receive the cup, the collar of which supports it in position. The precise bore of the sleeve is such as to provide the cup with an easy sliding fit.

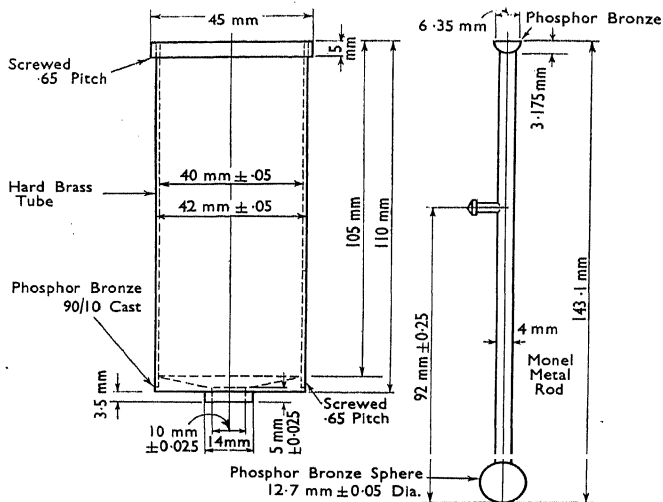


Fig. 15.—Viscosity of Refined Tar—Standard 10 mm Tar Cup and Valve therefor.

This sleeve is in turn surrounded by a cylinder with an easy sliding fit and provided with four vanes, the upper and lower portions of which are turned in opposite directions to form a stirring device. The cylinder is cut away between the vanes to facilitate heat transfer from the water in the bath to the tar cup; vertical grooves are provided on the inner surface of the cylinder to prevent the water in the bath from being carried into the tar cup if the stirring system be raised. A curved shield is fixed to the upper edge of the cylinder and extends to within about 5 mm of the walls of the water-bath.

This shield carries an insulated handle for rotating the stirrer, a support for a thermometer (see below), and a swivelled support for the valve. The two positions of this valve support are respectively (a) well clear of the tar cup and (b) extending over the tar cup so that the valve, when resting in the support, hangs vertically over and 16 mm \pm 1 above the orifice of the tar cup.

Three equidistant legs are riveted to the cylindrical wall of the bath and are of sufficient length to allow a standard 100 ml cylinder to be placed below the orifice of the cup.

In those laboratories where many samples are tested, it will be found advantageous to use a larger bath provided with a mechanical stirrer and thermostatic control. Several cylindrical sleeves will be fitted and so placed that the distances between them along the centre lines and between them and the side of the bath are about the same as for the single unit bath.

The water-bath may be adapted for electrical heating, provided adequate precaution is taken in construction to avoid undue local heating.

Standard thermometers having the range 15° to 45°C (Schedule No. T5a, page 333).

A receiver, not greater than 29 mm in internal diameter, with graduations at 20 ml, 25 ml and 75 ml capacities. A graduated 100 ml measuring cylinder complying with B.S. 604—1935: Graduated Measuring Cylinders meets these requirements.

Method:

The tar viscometer shall be so adjusted that the top of the tar cup is level; the water in the water-bath shall be brought to between 30.0° and 30.1°C inclusive, and maintained within these limits throughout its bulk for the duration of the test, the stirrer being gently rotated at frequent intervals or preferably continuously.

The tar cup of the viscometer shall be cleaned with a suitable solvent and thoroughly dried (see Note i). The material under examination (see Note ii) shall be warmed to 50°C and cooled, with stirring during both the heating and cooling processes. When the temperature has fallen to slightly above 30.0°C, the tar shall be poured into the tar cup until the levelling peg on the valve is just immersed when the latter is vertical.

A standard thermometer as specified shall be placed in the tar and used to stir the latter until the temperature is between

30.0° and 30.1°C inclusive. The thermometer shall then be suspended co-axially with the cup and with its bulb approximately at the geometric centre of the tar; for this purpose the 100 mm immersion mark on the thermometer shall be level with the top of the hemisphere on the upper end of the valve. The assembled apparatus shall be allowed to stand for five minutes during which period the thermometer reading must remain within ± 0.05 Centigrade degree of 30.0°C. The thermometer shall be withdrawn and any excess of tar removed so that the final level is on the centre line of the levelling peg when the valve is in the vertical position.

The receiver, containing 20 ml of mineral oil or of a 1 *per cent* *w/w* solution of soft soap, shall be placed under the orifice of the cup. The valve shall be suspended, by means of the hemisphere at the upper end, in the valve support, which shall have been brought immediately previously to its inner position. A stop watch shall be started when the reading in the cylinder is 25 ml and stopped when it is 75 ml; i.e. the time in seconds for running out 50 ml shall be noted. This figure shall be reported as the viscosity of the sample at 30.0°C (see Notes iii and iv).

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 4 *per cent* of the average of the several readings.

Notes:

(i) The tar cup is clearly a critical part of the viscometer, and special precautions must be observed in its treatment and use. Any cleaning process shall be of a gentle nature and non-corroding solvents such as light tar oils free from phenols are recommended rather than mechanical means such as dusters, which are liable to cause abrasion of the metal, especially at the orifice. If any material be used for rubbing the interior of the cup, it shall not be such as will disintegrate in use, leaving particles which may interfere with the free flow of the sample through the orifice.

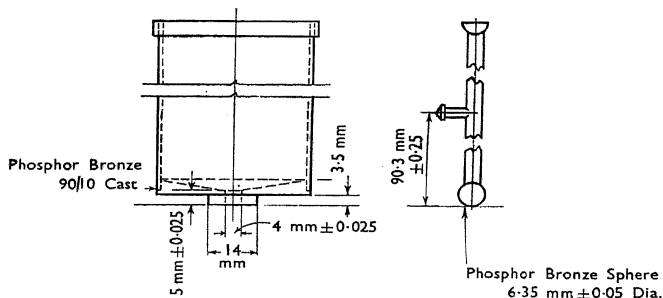
In addition to the above precautions, the orifice of the tar cup shall be tested at frequent intervals with a "go/no-go" gauge having appropriate diameters.

(ii) The presence of water, particularly in quantities under 1 *per cent*, has a marked effect on the viscosity, and the operator

must decide according to the end in view whether the viscosity of the sample as received or of the dehydrated sample shall be determined.

(iii) For convenience, the foregoing method has been described on the basis of conditions which are at present most commonly applied, but certain modifications are recognized as essential to meet various applications of the tar viscometer. Too low a viscosity reading gives rise to difficulties in the accurate measurement of the time of flow, while true stream-line flow through the orifice cannot be assumed if the time of flow is unduly long. The instrument must therefore be used in such circumstances that the efflux time lies within the range 10 to 140 seconds. To meet these conditions—

(a) A tar cup with an orifice $4\text{ mm} \pm 0.025$ in diameter is recognized as an alternative standard for the examination of particularly fluid tars, the cup being otherwise identical with the standard; this cup is subsequently referred to as the 4 mm



Dimensions and other details as for standard cup and valve, except for dimensions which are shewn above.

Fig. 16.—Viscosity of Refined Tar—4 mm Tar Cup and Valve therefor.

cup (see Fig. 16, above). If the alternative standard be used, the valve shall have a sphere $6.35\text{ mm} \pm 0.05$ in diameter; the distance from the base of the sphere to the levelling peg shall be $90.3\text{ mm} \pm 0.25$; in other respects the valve shall be identical with the standard valve (see Fig. 16).

(b) The instrument in its standard form or fitted with a 4 mm cup, may be used for thin tars at a lower temperature than 30.0°C , and in its standard form (i.e. fitted with a 10 mm cup) for especially thick tars at a higher temperature; this

latter modification is definitely required in certain standard specifications. Alternative appropriate standard thermometers to those specified (Schedule No. T5a) may be required in such cases and in the preliminary heating of the tar prior to filling the tar cup, the temperature shall be raised 20 Centigrade degrees above the temperature of the actual test.

It is essential that the details of any variation from the standard procedure, by the use of a 4 mm cup or by working at a temperature other than 30.0°C, be included in the analyst's report.

(iv) The results of tests made with the standard viscometer specified above, namely with a swivelled support for the valve and a 10 mm cup with the bottom conical in shape internally are the same within the limits of experimental error as those obtained with the viscometer specified in the 1929 edition of "Standard Methods."

(v) At a meeting of the International Tar Conference in 1937, the foregoing method was adopted in its entirety, but in certain countries the standard thermometer (graduated for 100 mm immersion) has since been replaced by a total immersion thermometer, no allowance being made for the exposed stem.

SERIAL No.: R.T. 3-38

Equi-viscous Temperature (E.V.T.)

(Standardization of Tar Products Tests Committee, *J. Soc. chem. Ind.*, 1937, 56, 422T.)

Definition:

The equi-viscous temperature (E.V.T.) of a tar is defined as the temperature in Centigrade degrees at which the viscosity of the tar is 50 seconds when determined according to the method described in Serial No. R.T. 2, page 97, using the standard 10 mm cup.

Notwithstanding the above definition, the E.V.T. when required in connexion with the sale and purchase of tar shall be understood to be that obtained by the appropriate part of the "Method" below.

Method:

PART A. TARS OF E.V.T. AT OR ABOVE 17.5°C

The viscosity of the tar shall be determined by the method described in Serial No. R.T. 2, page 97, using a standard 10 mm

cup at the temperature determined by the following circumstances:—

(a) If a specification which indicates the temperature of test is being followed, that temperature shall of course be used.

The E.V.T. shall be obtained from the viscosity and the temperature of test in Centigrade degrees by reference to Table 23, below, which shows the correction to be applied to the temperature of test to give the E.V.T. when the viscosity has been determined using the standard 10 mm cup.

TABLE 23: CORRECTIONS IN CENTIGRADE DEGREES TO BE APPLIED TO TEMPERATURES OF TEST, TO GIVE E.V.T.S OF TARS OF KNOWN VISCOSITY
STANDARD 10 MM CUP

Viscosity in seconds	0	1	2	3	4	5	6	7	8	9
10	-10.4	-9.8	-9.2	-8.7	-8.2	-7.7	-7.3	-6.9	-6.5	-6.1
20	-5.7	-5.4	-5.1	-4.8	-4.5	-4.3	-4.0	-3.8	-3.5	-3.3
30	-3.1	-2.9	-2.7	-2.5	-2.3	-2.2	-2.0	-1.9	-1.7	-1.5
40	-1.4	-1.2	-1.1	-0.9	-0.8	-0.6	-0.5	-0.4	-0.3	-0.1
50	0	+0.1	+0.2	+0.3	+0.5	+0.6	+0.7	+0.8	+0.9	+1.0
60	+1.1	+1.2	+1.3	+1.4	+1.5	+1.6	+1.7	+1.7	+1.8	+1.9
70	+2.0	+2.1	+2.2	+2.2	+2.3	+2.4	+2.5	+2.5	+2.6	+2.7
80	+2.8	+2.8	+2.9	+3.0	+3.0	+3.1	+3.1	+3.2	+3.3	+3.3
90	+3.4	+3.5	+3.5	+3.6	+3.6	+3.7	+3.7	+3.8	+3.9	+3.9
100	+4.0	+4.0	+4.1	+4.1	+4.2	+4.2	+4.3	+4.3	+4.4	+4.4
110	+4.5	+4.6	+4.6	+4.7	+4.7	+4.8	+4.8	+4.9	+4.9	+5.0
120	+5.0	+5.1	+5.1	+5.2	+5.2	+5.2	+5.3	+5.3	+5.4	+5.4
130	+5.5	+5.5	+5.5	+5.6	+5.6	+5.7	+5.7	+5.7	+5.8	+5.8
140	+5.9	+5.9	+6.0	+6.0	+6.0	+6.1	+6.1	+6.1	+6.2	+6.2

NOTE: That part of the table giving corrections for tars having viscosities between 33 and 75 seconds inclusive may alone be used in calculating the E.V.T. to be reported when no temperature of test is stated or implied by a tar specification. The remainder of the table will be useful in ranging tests.

(b) If a specification which does not indicate the temperature of test is being followed, or in the absence of a specification, the viscosity shall be determined at that temperature which is the multiple of 5 Centigrade degrees nearest to, or 2.5 Centigrade degrees above the expected E.V.T. If the temperature chosen be the correct one, the viscosity obtained will lie within the range 33 to 75 seconds; in this case the E.V.T. is obtained by reference to Table 23, above.

If the viscosity obtained be outside the range 33 to 75 seconds, the approximate E.V.T. shall be ascertained from the

table and the determination of the viscosity shall be repeated at the proper temperature. The E.V.T. shall be obtained from the result of the repeat determination by reference to the table.

The following example illustrates the procedure when a re-test is necessary:—

Example:

A tar tested at 35°C is found to have a viscosity of 92 seconds, which is outside the permissible range of 33 to 75 seconds. Table 23, page 105, gives a correction of +3.5 Centigrade degrees to be applied, making the E.V.T. approximately 38.5°C. The determination of the viscosity is therefore repeated at the temperature which is the multiple of 5 Centigrade degrees nearest to 38.5°C, i.e. at 40°C. At this temperature the viscosity is found to be 38 seconds. From Table 23, it is found that a correction of -1.7 Centigrade degrees is to be applied to the temperature of test. The E.V.T. is therefore $(40 - 1.7)^\circ\text{C} = 38.3^\circ\text{C}$.

PART B. TARS OF E.V.T. BELOW 17.5°C

The viscosity of the tar shall be determined by the method described in Serial No. R.T. 2, page 97, but using a 4 mm cup at the temperature determined by the following circumstances:—

(a) If a specification which indicates the temperature of test is being followed, that temperature shall of course be used.

The E.V.T. shall be obtained from the viscosity and the temperature of test in Centigrade degrees by reference to Table 24, opposite, which shews the correction to be applied to the temperature of test to give the E.V.T. when the viscosity has been determined using a 4 mm cup.

(b) If a specification which does not indicate the temperature of test is being followed, or in the absence of a specification, the viscosity shall be determined at that temperature which is the multiple of 5 Centigrade degrees nearest to, or 2.5 Centigrade degrees above the expected E.V.T. *plus* 20 Centigrade degrees. If the temperature chosen be the correct one, the viscosity obtained will lie within the range 61 to 106 seconds; in this case the E.V.T. is obtained by reference to Table 24, opposite.

If the viscosity obtained be outside the range 61 to 106 seconds, the approximate E.V.T. shall be ascertained from the table and the determination of the viscosity shall be repeated at the proper temperature. The E.V.T. shall be obtained from the result of the repeat determination by reference to Table 24.

The following example illustrates the procedure when a re-test is necessary:—

Example:

A tar tested at 30°C (with a 4 mm cup) is found to have a viscosity of 200 seconds, which is outside the permissible

TABLE 24: CORRECTIONS IN CENTIGRADE DEGREES TO BE APPLIED TO TEMPERATURES OF TEST, TO GIVE E.V.T.'S OF TARS OF KNOWN VISCOSITY
4 MM CUP

Viscosity in seconds	0	1	2	3	4	5	6	7	8	9
20	-33.9	-33.3	-32.8	-32.3	-31.8	-31.3	-30.9	-30.5	-30.1	-29.7
30	-29.4	-29.0	-28.7	-28.4	-28.1	-27.8	-27.5	-27.2	-27.0	-26.8
40	-26.6	-26.3	-26.1	-25.8	-25.6	-25.4	-25.2	-25.0	-24.8	-24.6
50	-24.4	-24.2	-24.0	-23.8	-23.7	-23.5	-23.4	-23.2	-23.1	-22.9
60	-22.7	-22.5	-22.4	-22.2	-22.1	-21.9	-21.8	-21.6	-21.5	-21.3
70	-21.2	-21.1	-21.0	-20.9	-20.8	-20.7	-20.6	-20.5	-20.4	-20.3
80	-20.2	-20.1	-20.0	-19.9	-19.7	-19.6	-19.5	-19.4	-19.3	-19.2
90	-19.1	-19.0	-18.9	-18.8	-18.7	-18.6	-18.5	-18.4	-18.4	-18.3
100	-18.2	-18.1	-18.0	-17.9	-17.8	-17.7	-17.6	-17.5	-17.4	-17.3
110	-17.2	-17.1	-17.1	-17.0	-16.9	-16.8	-16.8	-16.7	-16.7	-16.6
120	-16.6	-16.5	-16.4	-16.3	-16.2	-16.1	-16.1	-16.0	-16.0	-15.9
130	-15.8	-15.7	-15.7	-15.6	-15.6	-15.5	-15.5	-15.4	-15.4	-15.4
140	-15.4	-15.3	-15.3	-15.2	-15.2	-15.1	-15.1	-15.0	-15.0	-14.9
150	-14.9	-14.8	-14.7	-14.6	-14.6	-14.5	-14.5	-14.4	-14.4	-14.3
160	-14.3	-14.2	-14.2	-14.2	-14.1	-14.1	-14.1	-14.0	-14.0	-13.9
170	-13.9	-13.8	-13.8	-13.7	-13.7	-13.6	-13.6	-13.5	-13.5	-13.4
180	-13.4	-13.4	-13.3	-13.3	-13.3	-13.2	-13.2	-13.2	-13.1	-13.1
190	-13.0	-13.0	-12.9	-12.9	-12.8	-12.8	-12.8	-12.7	-12.7	-12.6
200	-12.6	-12.5	-12.5	-12.5	-12.4	-12.4	-12.4	-12.3	-12.3	-12.3

NOTE: That part of the table giving corrections for tars having viscosities between 61 and 106 seconds inclusive may alone be used in calculating the E.V.T. to be reported when no temperature of test is stated or implied by a tar specification. The remainder of the table will be useful in ranging tests.

range of 61 to 106 seconds. Table 24, above, gives a correction of -12.6 Centigrade degrees to be applied, making the E.V.T. approximately 17.4°C. The determination of the viscosity is therefore repeated at the temperature which is the multiple of 5 Centigrade degrees nearest to $(17.4 + 20)^\circ\text{C} = 37.4^\circ\text{C}$, i.e. at 35°C. At this temperature the viscosity is found to be 104 seconds. From Table 24, it is found that a

correction of -17.8 Centigrade degrees is to be applied to the temperature of test. The E.V.T. is therefore $(35 - 17.8)^{\circ}\text{C} = 17.2^{\circ}\text{C}$.

Limits of Accuracy:

The results of repeat determinations on portions of the same tar sample should fall within ± 0.2 Centigrade degree of the average of the several readings.

It should be noted by those engaged in scientific investigations as distinct from tests made in conjunction with the sale and purchase of tar that the E.V.T. determined as described under "Method" may differ from the true E.V.T.

In the case of tars of E.V.T. at or above 17.5°C , the E.V.T. determined according to part A of the method may differ from the true E.V.T. by an amount in general not exceeding 10 *per cent* of the difference between the temperature of test and the E.V.T. If it be required to know the true E.V.T., the viscosity should be determined at two temperatures, differing by at least 5 but not more than 10 Centigrade degrees and such that the viscosities are within the range 25 to 100 seconds. The value of the E.V.T. is calculated for each temperature of test by reference to Table 23, page 105. If the same value be obtained, it represents the true E.V.T. If the two values are not identical, the true E.V.T. is calculated by means of the following formula:—

$$\text{True E.V.T.} = T_1 - \frac{d_1}{d_2 - d_1} (T_2 - T_1)$$

where

T_1 and T_2 = the values of the E.V.T. calculated from the two test temperatures; and

d_1 and d_2 = the respective corrections applied to the temperatures of test to give the E.V.T.

If the temperature of test be above the E.V.T., the corresponding value of d is negative.

Example:

Temperature of test	40°C
Viscosity	30 seconds
Correction, i.e. d_1	-3.1 Centigrade degrees
∴ E.V.T., i.e. T_1	36.9°C

Temperature of test	35°C
Viscosity	73 seconds
Correction, i.e. d_2	+2.2 Centigrade degrees
∴ E.V.T., i.e. T_2	37.2°C

$$\begin{aligned}
 \therefore \text{True E.V.T.} &= T_1 - \frac{d_1}{d_2 - d_1} (T_2 - T_1) \\
 &= 36.9 - \frac{-3.1}{2.2 - (-3.1)} (37.2 - 36.9) \\
 &= 37.1^\circ\text{C}
 \end{aligned}$$

In the case of tars of E.V.T. below 17.5°C, the value of the E.V.T. obtained by part B of the method may differ from the true E.V.T. by an amount in general not exceeding ± 1.0 Centigrade degree. If it be required to know the true E.V.T., it is necessary to determine the viscosity by the method described in Serial No. R.T. 2, page 97, using the standard 10 mm cup at two temperatures sufficiently low to give viscosities within the range 25 to 100 seconds. The log of the viscosity is plotted against the log of the temperature in *Fahrenheit* degrees. The points are joined by a straight line and the log of the E.V.T. obtained by interpolation.

Notes:

(i) In certain circumstances it may be desired to use tables giving, for each temperature which is a multiple of 5 Centigrade degrees, the actual E.V.T. for each recorded viscosity. Such tables are easily constructed from the series of corrections given in Tables 23 and 24, pages 105 and 107.

(ii) Reprints of the Committee's paper "The Equi-viscous Temperature of Road Tars" (*J. Soc. chem. Ind.*, 1937, **56**, 422 π -427 π) are available, price 9d. post free, on application to the Committee at 166 Piccadilly, London, W.1.

SERIAL No.: R.T. 4-38

Water

Apparatus:

A glass or metal flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6a, page 349).

Method:

One hundred grams of the thoroughly mixed sample shall

be weighed into the flask and 100 ml of solvent naphtha (B.S. 479—1938, Part B) shall be added.

The flask shall be attached to the Dean and Stark condensing and collecting system. Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray tube (see page 351). Finally, the volume of water in the graduated tube shall be measured at laboratory temperature. Assuming the density 1.00 g/ml, the number of millilitres of water collected shall be reported as the percentage by weight of water in the sample.

SERIAL NO.: R.T. 5—38

Distillation

Apparatus:

A standard distillation flask (Schedule No. F8, page 365).

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333), so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S3, page 373) fitted with a hard asbestos or silica board having a central hole 110 mm in diameter; the flask shall be placed in position and pressed down so as to close completely the hole in the board.

A standard air condenser (Schedule No. C3, page 370). The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a clamp to hold the condenser.

A weighed standard 25 ml Crow receiver (Schedule No. V3, page 341).

Weighed standard 100 ml Crow receivers (Schedule No. V1, page 341).

A large size Bunsen burner.

Material for Test:

If the water content of the sample as determined by the method described in Serial No. R.T. 4, page 109, be not greater than 0.5 *per cent*, the tar as received shall be used for the following test.

If the water content of the sample as so determined should exceed 0.5 *per cent*, about 1,000 ml of the sample shall be distilled until the water has come over. Any oil which appears in the distillate shall be separated and returned to the flask when the tar is sufficiently cool. After thorough mixing, 750 g of the contents of the flask shall be taken for distillation as described below.

Method:

750 g \pm 0.5 of the material for test (see above) shall be weighed into the weighed distillation flask and the apparatus assembled, using the 25 ml Crow receiver to collect the first fraction. This and subsequent receivers shall be placed so that the condensate will flow down the side. Heat shall be applied at such a rate that the thermometer indicates 200°C within 35 minutes \pm 5 from the start (see Note i). As soon as oil commences to distil, the flame shall be adjusted so that (a) 5 ml of distillate collect in at least 54 but not more than 66 seconds. This distillation rate (which corresponds to 5 ml \pm 0.5 per minute) shall be strictly maintained throughout the remainder of the distillation, except that (b) the collection of the first 5 ml in any receiver may extend over 75 seconds as the upper limit, and (c) if the collection of any 5 ml extends over less than 54 or more than 66 seconds, the collection of 15 ml including that 5 ml shall extend over at least 165 but not more than 195 seconds. If in any test the foregoing conditions are not met, the test shall be discarded and another test carried out with a further portion of the sample (see Note ii).

The specified distillation rate corresponds approximately to 90 drops per minute or 3 drops in each 2 seconds, but this must only be taken as a guide and graduated receivers are specified for the collection of the distillate so that the time taken to collect each 5 ml may be kept under close observation. A record of these times shall be made throughout the test so that compliance with the distillation requirements may be subsequently checked.

When the thermometer indicates 200°C (see Note iii), the 25 ml Crow receiver shall be replaced by one of the 100 ml Crow

receivers which has been warmed immediately before use, so that naphthalene is prevented from solidifying on its inner wall and so interfering with the checking of the distillation rate.

The other fractions shall be taken at 270°C and 300°C, or such other temperatures as may be specified; they shall be collected in the 100 ml Crow receivers, the receivers being changed without interrupting the distillation except that the Bunsen flame shall be extinguished immediately the highest specified temperature is reached. The final fraction shall include all oil which drains from the condenser within five minutes after the flame has been extinguished.

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

The weight of the distillate to 200°C shall be ascertained in grams; the volume of the water shall be noted and, assuming a density of 1.00 g/ml, shall be recorded as the weight of water in grams. The weight of the light oil in the fraction to 200°C shall be found by difference. The weights of the other fractions shall also be determined, while in addition the volumes of the fractions "up to 200°C" and "200° to 270°C" shall be noted for use in connexion with the determination of phenols (Serial No. R.T. 6, opposite).

The flask shall remain with the cork and thermometer in position for one hour after the flame has been extinguished. The flask and residue shall then be weighed, after which the contents of the flask shall be mixed thoroughly by shaking and about half poured out. The remainder shall be again well shaken and examined, if necessary, by the Ring and Ball test described in Serial No. P.T. 4, page 317, the warm material being poured directly from the flask into the pitch rings.

The distillation test and the examination of the residue shall be carried out in duplicate; the separate results and the average shall be reported as percentages by weight on the sample.

Notes:

(i) When examining tars which are liable to froth on heating, it may be found that the temperature cannot satisfactorily be brought to 200°C within the maximum specified period of $(35 + 5 =) 40$ minutes. Only in the testing of such tars may the preliminary period of heating be extended and then only just sufficiently for the purpose; *in such an event the time taken to reach 200°C shall be recorded in the analyst's report.*

(ii) A water gauge connected to the gas supply between the tap and the burner is useful as a guide for the control of the flame and thereby the distillation rate.

(iii) The specified temperatures shall not be corrected for the effect of barometric pressure but the latter figure shall be recorded. Any test required for referee purposes shall, however, be carried out when the barometric pressure is within the range $760 \text{ mm} \pm 5$; if this be impracticable, the specified temperatures shall be corrected according to the instructions on pages 64 to 69.

SERIAL No.: R.T. 6-38

Phenols

General:

The phenols are extracted by caustic soda and the soda solution is boiled to remove neutral oils and bases; the phenols are liberated by the addition of hydrochloric acid and their volume is measured.

Method:

The whole of the oil distilling to 270°C in the distillation test (Serial No. R.T. 5, page 110), warmed if necessary until completely liquid, shall be transferred to a stoppered separating funnel of appropriate capacity. An equal volume of a 10 *per cent w/w* clear aqueous caustic soda solution previously heated to between 40° and 70°C shall be used to rinse the receivers from which the distillates were transferred and added to the latter in the separating funnel. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement, the alkaline layer shall be run into a beaker. The upper layer shall be agitated for five minutes with a further quantity of the hot caustic soda solution, equal to one quarter of the volume of the distillate which is being washed, and with further successive quantities of the hot caustic soda solution, each equal in volume to the last, until all the phenols have been removed. This can be ascertained by *slightly* acidifying the last washings, using concentrated hydrochloric acid, and examining for separated phenols. After settlement, the alkaline layer shall in each case be added to the first soda washing. A large excess of soda must be avoided but some excess must be maintained; as a rough guide, it may be assumed that 25 ml of caustic soda solution of the strength indicated are

sufficient to remove about 5 ml of phenols. During the washing processes, the contents of the funnel shall be kept liquid, immersing the funnel if necessary in warm water at between 40° and 70°C.

The soda washings, including any acidified for the purpose of checking the washing procedure, shall be combined. The combined washings shall be alkaline; they shall be boiled vigorously for twenty minutes; fragments of porous or other suitable inert material may be used to prevent bumping.

The soda washings shall be cooled and, if clear, shall be transferred direct to the phenols flask (Schedule No. V5, page 347) (see Note i). If the solution be not clear, it shall be filtered through glass wool previously moistened with saturated aqueous sodium chloride solution; the filtrate shall be collected in the phenols flask; the glass wool shall be washed with 25 ml of saturated sodium chloride solution which shall be added to the filtered soda washings. Methyl orange indicator solution shall be added, followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the contents of the flask shall be kept cool by immersing the flask from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a *few* particles undissolved; the phenols shall be brought into the graduated portion of the flask by adding saturated sodium chloride solution.

After adequate time for settlement, preferably overnight, the volume of phenols shall be noted and the percentage by volume of the phenols in the original sample of tar shall be calculated and reported (see Notes ii and iii).

Notes:

(i) If it be expected that the tar contains $3\frac{1}{2}$ *per cent* or more of phenols, the boiled soda washings (after filtration through glass wool if necessary) shall be divided about evenly between two phenols flasks, the subsequent specified procedure being carried out on the contents of each flask and the volumes of phenols recovered being totalled.

(ii) The phenols as determined always contain water which has not been derived from the distillate examined.

(iii) When the percentage of phenols is required by weight, the assumed density 1.04 g/ml shall be used for calculation.

SERIAL No.: R.T. 7-38

Naphthalene

It is to be understood that this determination is only in respect of the naphthalene deposited at 15.5°C from solution in the total distillate to 270°C.

Method:

The whole of the oil distilling to 270°C in the distillation test (Serial No. R.T. 5, page 110) which was washed for phenols in the method described in Serial No. R.T. 6, page 113, shall be cooled to $15.5^{\circ}\text{C} \pm 0.5$ Centigrade degree and maintained within those limits of temperature for two hours with occasional stirring.

The material shall then be transferred to a cold Buchner funnel fitted with a rapid filter paper, and as much oil as possible removed rapidly by a filter pump. The crude naphthalene shall be placed between sheets of blotting paper and submitted to pressure in a hand screw-press. The oily margins of the cake shall be removed and separately pressed.

The operations, starting with the transfer to the Buchner funnel to the preliminary pressing, shall be conducted with the utmost rapidity.

The pressed naphthalene shall be weighed and the percentage by weight in the tar itself shall be calculated and reported. The "corrected wet crystallizing-point" of the pressed naphthalene shall be determined by the method described in Serial No. C.N. 2, page 248. The crystallizing-point shall be at least 70°C.

SERIAL No.: R.T. 8-38

Insoluble Matter ("Free Carbon") and Ash**Method:**

Two grams of the thoroughly mixed sample shall be warmed on the water bath and mixed intimately in a 200 ml beaker with 100 ml of hot pure toluole (B.S. 805—1938, Part A) (see Notes i and ii). After settlement for twenty minutes with the beaker standing on the water-bath, the supernatant liquid shall be decanted cautiously, avoiding disturbance of the sediment, either (a) through balanced filter papers (e.g. Whatman No. 5) about 150 mm in diameter, or (b) through a weighed Gooch crucible prepared with asbestos and used under vacuum.

If filter papers are used, they must be heated to a temperature between 95° and 110°C and reduced to equal weight by removing

the apex of the heavier paper; the uncut paper is used inside the originally heavier paper. The separated material is retained by the inner paper, but both of them are equally subjected to any action exerted by the toluole.

Washing shall be effected repeatedly *by decantation* until 300 ml of the toluole have been used. The residue shall be transferred to the filter and the washing continued until altogether 500 ml have been used. The residue shall be dried in an oven at a temperature between 95° and 110°C until it is of constant weight. The residue shall be incinerated, the weight of the ash deducted (after allowing for that in the filter paper) and the percentage by weight of ash-free "free carbon" and of ash in the sample shall be calculated and reported.

Notes:

(i) The temperature of the toluole throughout the determination must be between 90° and 100°C.

(ii) *If the above method be varied by the use of any solvent other than hot pure toluole to the above-mentioned specification the result shall be recorded as "matter insoluble in ——" and not as "free carbon."*

4. CRUDE BENZOLE

Applicability:

The tests in this section are applicable to crude benzole and crude low temperature spirit, and to crude light products resulting from the distillation of crude tar and having a distillation range similar to that of crude benzole.

The application of these methods to products containing substantial amounts of paraffins must be made with circumspection.

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

It will be clear from the subsequent instructions that the quantity required for test depends upon the extent to which the examination will proceed. At least one pint (about 600 ml) shall be supplied if preliminary tests are alone to be made and at least one quart (about 1,200 ml) if the full scheme of tests covered by this section is to be followed and if the material be of normal quality. If material of special quality is to be examined, the quantity of sample to be supplied must be left to the discretion of the analyst.

Preliminary Treatment of the Sample:

Any separated water shall be removed and its quantity noted, before any portion of the sample is withdrawn for test.

The examination of crude benzole is conveniently prescribed under two heads, viz. Preliminary Tests and Commercial Analysis, as below.

PRELIMINARY TESTS

SERIAL NO.: C.B. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity), or Serial No. G.P. 1, page 31 (density)).

Distillation

General:

In Great Britain, at present, it is still customary to distil 100 ml of the sample in a retort, with the bulb of the thermometer in the liquid. The volume of the distillate at 120°C (stop-point) is taken as the basis for the regulation of the price of the material.

The Committee has again fully considered the question of standardizing such a retort test, but must confirm its previous view, based on extensive practical investigation prior to the publication of the 1929 edition of "Standard Methods," that it is impracticable to evolve a satisfactory standard test on the above mentioned lines. Moreover, the retort test for benzole is now considered to be unsatisfactory as a criterion of quality, and this opinion is supported by a majority of the producers of benzole. The following method, based on a test devised by the National Benzole Co. Ltd and reproduced by permission, may with advantage be substituted for the retort test.

Apparatus:

A bolt head flask having the following dimensions: Bulb capacity: 200 ml ± 10 ; length of neck: 33 mm ± 3 ; internal diameter of neck: 34 mm ± 2 .

A standard 8-bulb pear column (Schedule No. A1, page 370).

A standard thermometer having the range 50° to 210°C (Schedule No. T9a, page 333).

A standard Liebig condenser with bent end (Schedule No. C1, page 369).

A standard 100 ml Crow receiver (Schedule No. V1, page 341).

A standard draught screen (Schedule No. S2, page 373).

A Bunsen burner, the flame of which shall be entirely non-luminous and as small as is conveniently possible.

The column shall be fitted to the flask by means of a cork, the bottom of the column extending 40 mm below the top of the neck of the flask. The flask and column shall be set vertically and supported by means of a clamp at the neck and not from the bottom. The side tube of the column shall be fitted to the condenser by means of a cork and shall extend at least 50 mm beyond the cork. The thermometer shall be so fitted in the top of the column that the bottom of the

capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366). The draught screen shall be placed on the bench concentrically with the burner. No gauze or asbestos sheet shall be interposed between the flask and the burner. The column shall not be lagged in any way.

Method:

One hundred millilitres of the sample (see Note i) shall be measured into the 100 ml receiver and its temperature shall be noted. It shall be transferred to the distillation flask, the contents of the receiver being allowed to drain into the flask for fifteen seconds.

Twenty millilitres of prepared creosote oil (see Note ii) shall be measured into the same receiver without rinsing or drying and shall also be transferred to the flask, allowing the receiver to drain into the flask for fifteen seconds. A fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping. The apparatus shall be assembled as described under "Apparatus," the 100 ml receiver being used after cleaning and drying, to receive the distillate; the position of the receiver shall be so adjusted that the condensate flows down its side.

The contents of the flask shall be distilled at the rate of 5 ml per minute (about two drops per second). The readings of volume and temperature (see under "Temperature Corrections" below) shall be made according to the requirements specified, the measurements being taken at running-points or stop-points as required (see Note iii).

In the absence of other instructions, the distillate to 160°C (stop-point) shall be collected and its volume noted.

Temperature Corrections:

In the absence of other instructions, the boiling-points shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69.

Notes:

(i) If water should *accumulate* during distillation, it shall be measured, but the test shall be discarded and a further portion of the sample shall be dried as follows:—

About 100 g of finely powdered plaster of Paris (calcium sulphate hemihydrate) shall be placed in a dry wide mouthed

stoppered glass bottle having a capacity of about 450 ml and an external diameter of from 50 to 60 mm. About 250 ml of the sample shall be introduced and the bottle stoppered. The bottle shall be well shaken for ten minutes and allowed to stand. One hundred millilitres of the dried and settled sample shall be decanted into the Crow receiver for the test.

In these circumstances, the report shall indicate that the test was carried out on the dried sample, and due allowance shall be made for water, the amount of which shall be reported separately.

(ii) The creosote oil shall be prepared by fractionating a quantity of washed creosote oil (i.e. creosote oil containing 3 per cent or less of phenols) through the standard column (Schedule No. A1, page 370) to 190°C (stop-point). The residue in the flask at that temperature shall be cooled to 15·5°C and kept within 0·5 Centigrade degree of that temperature for at least two hours. Twenty millilitres of the fluid portion of the cooled residue shall be decanted into the Crow receiver as described under "Method" above.

(iii) The temperature and flow of the cooling water in the condenser shall be such that the temperature of the distillate is maintained within ± 5 Centigrade degrees of that at which the sample was measured for test. When this is impracticable, running-point observations cannot be regarded as accurate.

SERIAL No.: C.B. 3-38

Total Sulphur

Material for Test:

In the absence of other instructions, the distillate to 160°C (stop-point) in the distillation test (Serial No. C.B. 2, page 118) shall be used after the following preliminary treatment:—

A portion of the distillate to 160°C, the specific gravity of which has been determined, shall be shaken for half a minute with 10 per cent by volume of 10 per cent w/w clear aqueous caustic soda solution. It shall be separated from the soda layer, washed with water until free from alkali and finally dried over plaster of Paris (calcium sulphate hemihydrate).

Occasionally, a determination of the amount of total sulphur in the original sample or in the separate fractions is required; *if such a determination be performed, the report on the test shall make that clear.*

Method:

The total sulphur content of the washed and dried material for test shall be determined according to the method described in Serial No. L.B. 14, page 164, and the result reported as the percentage by weight on the material taken for test.

SERIAL No.: C.B. 4-38

Carbon Disulphide**Material for Test:**

In the absence of other instructions, the distillate to 160°C (stop-point) in the distillation test (Serial No. C.B. 2, page 118) shall be used after the following preliminary treatment:—

A portion of the distillate to 160°C, the specific gravity of which has been determined, shall be shaken for half a minute with 10 *per cent* by volume of 10 *per cent* w/w clear aqueous caustic soda solution. It shall be separated from the soda layer, washed with water until free from alkali and finally dried over plaster of Paris (calcium sulphate hemihydrate).

Occasionally, a determination of the amount of carbon disulphide in the original sample is required; *if such a determination be performed, the report on the test shall make that clear.*

Method:

The carbon disulphide content of the washed and dried material for test shall be determined according to the method described in Serial No. L.B. 16, page 171, and the result reported as the percentage by weight on the material taken for test.

COMMERCIAL ANALYSIS

SERIAL No.: C.B. 5-38

Loss on Acid-washing**Preliminary Treatment of the Sample:**

The sample will in the majority of cases be sufficiently clean for examination without preliminary distillation, unless the sample contains more than 10 *per cent* distilling above 190°C. In that case a high percentage of wash oil or creosote oil is indicated and 500 ml of the sample shall be distilled in the same manner as is described in Serial No. C.B. 6b, page 124, the distillation being carried to 190°C (stop-point). The distillate

shall be examined as described below; the residue shall be measured at laboratory temperature and reported as wash oil or creosote oil.

Method:

A convenient quantity, preferably 500 ml, of the sample or the distillate to 190°C as the case may be (see above) shall be washed with sufficient 10 *per cent w/w clear* aqueous caustic soda solution to remove all the phenols and hydrogen sulphide. Pyridine bases shall then be removed by washing with 25 *per cent w/w* sulphuric acid. In the case of samples already free from phenols and hydrogen sulphide, and/or free from pyridine bases, this part of the method may be modified appropriately.

The prepared sample (see above), free from visible water, shall be measured at laboratory temperature into a stoppered separating funnel and 5 *per cent* of its volume of 95 ± 0.5 *per cent w/w* sulphuric acid shall be added (see Note i). If the proportion of unsaturated hydrocarbons be high, great caution is necessary during this addition and the acid must be introduced in small portions, with vigorous cooling after each addition.

The contents of the separating funnel shall be shaken; care is necessary at first, otherwise the mixture may become uncontrollably hot and burst the vessel or force out the stopper. Any *undue* rise in temperature shall be checked by cooling under running water from time to time. At intervals during the shaking period the pressure may be released by inverting the separating funnel and opening the tap. Shaking shall be continued for ten minutes and shall be vigorous as soon as the temperature is found to be under control. The mixture shall be allowed to settle for fifteen minutes and 10 to 20 ml of water shall be introduced down the side of the funnel in order to create as little disturbance as possible. Within a minute or two this forms a visible layer between the dark acid and the spirit, and enables the acid to be removed without risking loss of spirit. The acid and water layers shall be run off and discarded. A further 10 to 20 ml of water shall be introduced and worked round the sides of the separating funnel to remove any acid tar there deposited. Agitation must be avoided as it may result in the formation of an obstinate emulsion. After settlement, this washing shall also be run off and discarded.

Clear aqueous caustic soda solution (e.g. 10 *per cent* by volume of a 20 *per cent w/w* solution) shall be added and agitated gently with the spirit, avoiding the formation of an

emulsion, until the spirit changes in colour to yellow or brown. This change may require several minutes and the gentle agitation shall be continued until no further colour change is observed. The mixture shall be allowed to settle until the aqueous portion has separated. The volume of the washed spirit shall be measured at laboratory temperature (see Note ii).

The loss on acid-washing in millilitres is the difference in volume of the sample before and after treatment, *plus* the number of millilitres of water recovered during the subsequent fractionation (see Serial No. C.B. 6, below); from this, the percentage loss on acid-washing shall be calculated and reported.

Notes:

(i) The above procedure, which is an essential treatment preliminary to the Colman and Yeoman test (see Serial No. C.B. 6a, below), represents drastic acid-washing treatment and is intended to be correlated with the manufacture of pure products.

(ii) In order to ascertain whether the removal of the unsaturated hydrocarbons has been substantially complete, the following test may be applied:—

Ten millilitres of Normal sulphuric acid shall be placed in a small stoppered cylinder together with two millilitres of the washed spirit; one millilitre of 0.5N bromide-bromate solution (see Serial No. L.B. 12, page 160, for a method of preparation of this solution) shall be added and the whole shaken vigorously for one minute. If the colour be discharged, the washed material must be regarded as unsuitable for examination by such tests as Serial Nos L.B. 21, 22, 23a, 23b, 24, 25 and 27, pages 180 *et seq.*

SERIAL No.: C.B. 6–38

Fractionation

General:

The subsequent analysis shall be carried out according to one or other of the alternatives given below (Serial Nos 6a and 6b), the selection being determined by the nature of the information required.

SERIAL No.: C.B. 6a–38

Determination of Pure Products

Method:

The Colman and Yeoman test shall be carried out as described in Serial No. L.B. 27, page 192, except that the whole of the

acid-washed material obtained in Serial No. C.B. 5, page 121, shall be used for test instead of the 500 ml of sample prescribed in Serial No. L.B. 27.

The following shall be reported as percentages by volume on the original crude sample freed from any separated water:—

Carbon disulphide
Paraffins
Benzene
Toluene
Xylenes
Heavy naphtha
Loss on acid-washing
Wash oil or creosote oil

the last mentioned being the residue in the fractionation process of the Colman and Yeoman test *plus* the residue in the preliminary fractionation in Serial No. C.B. 5, if that fractionation has been carried out.

SERIAL No.: C.B. 6b-38

Determination of Commercial Fractions

General:

If it be decided that the Colman and Yeoman test is unnecessary, the analysis being required, not in terms of pure products, but on the basis of the contents of commercial benzole, commercial toluole, solvent naphtha, etc., the following fractionation test shall be carried out.

Apparatus:

A bolt head flask of about 650 ml bulb capacity fitted with a standard 12-bulb pear column (Schedule No. A2, page 370), the side arm of which is connected to a standard Liebig condenser with bent end (Schedule No. C1, page 369). The flask is supported by means of a clamp at the neck and no gauze or asbestos sheet is interposed between it and the burner. The column shall not be lagged in any way.

A standard thermometer having the range 50° to 210°C (Schedule No. T9a, page 333), so fitted in the top of the column that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S2, page 373).

A number of suitable graduated receivers.
A Bunsen burner.

Method:

The whole of the acid-washed material obtained in Serial No. C.B. 5, page 121, shall be transferred to the flask. A fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping and the apparatus assembled with the column vertical. The contents of the flask shall be distilled at a rate of 4 to 5 ml per minute (about two drops per second), the graduated receivers being used to collect the distillates (see Note). Fractions shall be taken as shewn below:—

Up to 100°C	Commercial benzole
100° to 120°C	Commercial toluole
120° to 160°C	Solvent naphtha
160° to 190°C	Heavy naphtha

each temperature being a running-point except 190°C, which shall be a stop-point (see under "Temperature Corrections" below). The distillates shall be brought to laboratory temperature and measured. The results provide an approximation to the content of the several commercial fractions shewn above. The residue in the flask shall be transferred to a graduated receiver, measured at laboratory temperature and reported as wash oil or creosote oil.

The specific gravity of each distillate may be determined to give some indication of the paraffins content of the respective fractions. The distillates may additionally be subjected to any of the appropriate tests for refined lower boiling products (see pages 141 *et seq.*).

The following shall be reported as percentages by volume on the original crude sample freed from any separated water:—

Commercial benzole
Commercial toluole
Solvent naphtha
Heavy naphtha
Loss on acid-washing
Wash oil or creosote oil

the last mentioned being the residue in the foregoing fractionation *plus* the residue in the preliminary fractionation in Serial No. C.B. 5, if that fractionation has been carried out.

Temperature Corrections:

The boiling-points shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69.

Note:

If 5 ml or more of water collect in the first receiver, the distillation shall be continued into that receiver until water ceases to come over, when the fractionation shall be stopped. The distillate shall be allowed to settle and the volume of water shall be noted and included as part of the loss on acid-washing in Serial No. C.B. 5, page 121. The whole of the contents of the receiver shall be transferred to a separating funnel and allowed to settle. The aqueous portion of the distillate shall be separated and the remaining oily distillate returned to the flask when cool. The distillation shall be restarted and fractions taken as described under "Method."

5. LIGHT AND MIDDLE OILS

Applicability:

The tests in this section are applicable to light oil and to middle or carbolic oil.

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

It will be clear from the subsequent instructions that the quantity required for test depends upon the extent to which the examination will proceed. At least one pint (about 600 ml) shall be supplied if preliminary tests are alone to be made and at least one quart (about 1,200 ml) if the full scheme of tests covered by this section is to be followed.

Preliminary Treatment of the Sample:

Any separated aqueous liquor shall be removed and its quantity noted, before any portion of the sample is withdrawn for test.

The sample, freed from any separated water as above mentioned, shall be thoroughly mixed immediately before any portion is withdrawn for testing. If it should contain separated soluble solid constituents, the container shall be immersed in a water-bath at a temperature just sufficient to dissolve them and portions for test shall be taken from the warmed mixed sample. No source of intense heat shall be used to warm the sample.

The examination of light and middle oils usually comprises a series of preliminary tests which may or may not be followed by a commercial analysis, according to the end in view.

PRELIMINARY TESTS

The preliminary tests usually consist of the following which shall be carried out according to the methods indicated:—

Specific gravity	L.M. 1
Water	L.M. 2
Distillation	L.M. 3
Phenols	L.M. 4

The report on the foregoing should be supplemented by a statement on the general physical appearance of the sample, including reference to the presence or absence of tarry matter, the extent of deposition of solid matter at laboratory temperature and any other special features which may be apparent or which, in the analyst's knowledge, will be required.

SERIAL NO.: L.M. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47, and Note ii on page 55 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL NO.: L.M. 2-38

Water

Apparatus:

A 100 ml graduated receiver.

A glass flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6 or V6a, pages 349 to 356, according as the sample is expected to contain more or less than 1.5 *per cent* of water).

Method:

One hundred millilitres of the thoroughly mixed sample shall be measured in the graduated receiver at laboratory temperature or the lowest temperature of complete liquidity if that be above laboratory temperature, and transferred to the flask which shall then be attached to the Dean and Stark condensing and collecting system (see Note). Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray tube (see page 351). Finally, the number of millilitres of water in the graduated tube shall be noted at the temperature

at which the sample was measured and shall be reported as the percentage by volume of water in the sample.

Note:

If the sample should not distil smoothly, 20 ml of solvent naphtha (B.S. 479—1938, Part B) may be added to the sample in the flask prior to distillation.

SERIAL No.: L.M. 3-38

Distillation

Apparatus:

A standard distillation flask (Schedule No. F4, page 365), held in the vertical position by means of a clamp at the extreme upper end of the neck.

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333), so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S3, page 373), from which the shelf has been removed.

A standard Liebig condenser with bent end (Schedule No. C1, page 369). The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A standard air condenser (Schedule No. C3, page 370).

A retort stand provided with a clamp to hold the condenser.

A standard 100 ml Crow receiver (Schedule No. V1, page 341).

A Bunsen burner, the flame of which shall be entirely non-luminous and as small as is conveniently possible.

Method:

One hundred millilitres of the sample (see Note), warmed if necessary until completely liquid, shall be measured into the 100 ml receiver and its temperature noted. The contents of the receiver shall be transferred to the distillation flask, the receiver being allowed to drain for fifteen seconds into the flask; a fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping. The apparatus shall be assembled, using the Liebig condenser only.

Distillation shall be carried out at the rate of 5 ml per minute (about two drops per second), the volumes of distillate at running-points and/or stop-points being taken as required. The Liebig condenser shall be replaced by the air condenser

at 200°C (stop-point), the distillation being resumed and running-points and/or stop-points being recorded as required until 95 ml of distillate have been collected.

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

Note:

If water should *accumulate* during distillation, it shall be measured, but the test shall be discarded and a further portion of the sample dried as follows:—

About 100 g of finely powdered plaster of Paris (calcium sulphate hemihydrate) shall be placed in a dry wide mouthed stoppered glass bottle having a capacity of about 450 ml and an external diameter of from 50 to 60 mm. About 250 ml of the sample shall be introduced and the bottle stoppered. The bottle shall be well shaken for ten minutes and allowed to stand. One hundred millilitres of the dried and settled sample shall be decanted into the 100 ml receiver for the test.

In these circumstances, the report shall indicate that the test was carried out on the dried sample, and due allowance shall be made for water, the amount of which shall be reported separately.

SERIAL No.: L.M. 4-38

Phenols

General:

The phenols are extracted with caustic soda and the soda solution is boiled to remove neutral oils and bases; the phenols are liberated by the addition of hydrochloric acid and their volume is measured.

Method:

The total distillate in the distillation test (Serial No. L.M. 3, page 129), warmed if necessary until completely liquid, shall be transferred to a stoppered separating funnel of about 250 ml capacity. Fifty millilitres (see Notes i and ii) of a 10 per cent w/w clear aqueous caustic soda solution, measured in the receiver from which the distillate was transferred, shall be added. If the distillate should contain naphthalene which tends to separate, both the distillate and the caustic soda solution shall be warmed just sufficiently to maintain the naphthalene in solution. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement, the alkaline layer shall be run into a beaker. The upper layer shall be agitated for five minutes with a further 25 ml of the caustic soda solution and with further successive quantities each of 10 ml of the caustic soda solution until all the phenols have been removed. This can be ascertained by *slightly* acidifying the last washings, using concentrated hydrochloric acid, and examining for separated phenols. After settlement, the alkaline layer shall in each case be added to the first soda washing. A large excess of soda must be avoided but some excess must be maintained; as a rough guide it may be assumed that 5 ml of caustic soda solution of the strength indicated are sufficient to remove about one millilitre of phenols. During the washing processes, the contents of the funnel shall be kept liquid, immersing the funnel in warm water if necessary.

The soda washings, including any acidified for the purpose of checking the washing procedure, shall be combined. The combined washings shall be alkaline; they shall be boiled vigorously for ten minutes, the initial volume being roughly maintained by the addition of distilled water; fragments of porous or other suitable inert material may be used to prevent bumping. The soda washings shall be cooled to laboratory temperature and if clear, shall be transferred direct to the phenols flask (Schedule No. V5, page 347; see Notes i and ii). If the solution be not clear, it shall be filtered through glass wool previously moistened with saturated aqueous sodium chloride solution; the filtrate shall be collected in the phenols flask; the glass wool shall be washed with 25 ml of saturated sodium chloride solution which shall be added to the filtered soda washings. Methyl orange indicator solution shall be added, followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the contents of the flask shall be kept cool by immersing the flask from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a *few* particles undissolved; the phenols shall be brought into the graduated portion of the flask by adding saturated sodium chloride solution. After adequate time for settlement, preferably overnight, the volume of phenols shall be read at the temperature at which the sample was measured for the distillation test (Serial No. L.M. 3, page 129) and shall be reported as "wet crude phenols" (see Note iii).

Notes:

(i) In the case of samples containing only small amounts of phenols, it is preferable to use half the specified volumes of caustic soda solution for the successive washings and to collect the soda washings (after filtration through the glass wool if necessary) in the alternative phenols flask, Schedule No. V5a, page 347.

(ii) If it be expected that the sample contains 25 *per cent* or more of phenols, the volumes of caustic soda solution shall be appropriately increased. The boiled soda washings (after filtration through the glass wool if necessary) shall be divided about evenly between two phenols flasks, the subsequent specified procedure being carried out on the contents of each flask and the volumes of phenols recovered being totalled.

(iii) The crude phenols as determined always contain water which has not been derived from the oil and they correspond to the "wet crude phenols" obtained in Serial No. L.M. 5a, page 133.

SERIAL No.: L.M. 5-38

COMMERCIAL ANALYSIS**General:**

The commercial analysis of light and middle oils, comprising the isolation and, if desired, examination of the following products, shall be carried out according to the methods indicated:—

Phenols	L.M. 5a
Pyridine bases	L.M. 5b
Crude naphthalene	L.M. 5c
Loss on acid-washing; refined naphthas, and neutral oils	L.M. 5d

If phenols or pyridine bases are known to be absent, the corresponding treatment may be omitted.

Preliminary Treatment of the Sample:

If the sample be clean enough to give good separation with caustic soda solution, the crude phenols may be immediately extracted as described in Serial No. L.M. 5a, opposite. Otherwise, the sample shall be submitted to distillation as described below.

The apparatus described in Serial No. L.M. 3, page 129, shall be used, except that the flask therein specified (Schedule No.

F4) shall be replaced by a larger one of suitable size (see below), while the receiver shall be correspondingly larger.

A suitable quantity of the sample, warmed if necessary until completely liquid, shall be measured and its temperature recorded; the amount of sample will be determined by the extent to which the analysis is to be carried; the distillation of 500 ml will usually provide sufficient material for a complete examination. The measured portion of the sample shall be transferred to the distillation flask, allowing fifteen seconds for draining, and the apparatus assembled, using the Liebig condenser only. The distillation shall be carried to 200°C (stop-point) when the Liebig condenser shall be replaced by the air condenser. The distillation shall be resumed and continued into the same receiver until 95 *per cent* of the sample has distilled.

If no preliminary distillation be required, the amount of sample for the subsequent tests will equally depend on the extent to which the analysis is to be carried; here also, 500 ml will usually be sufficient for a complete examination.

SERIAL No.: L.M. 5a-38

Phenols

(REMOVAL AND RECOVERY)

General:

The object of the following procedure is to extract the phenols from the sample in order to obtain (a) sufficient for examination by the methods described under "Carbolic and Cresylic Acids," pages 203 *et seq.*, and/or (b) an oil free from phenols for examination ultimately by the methods described under "Refined Lower Boiling Products," pages 141 *et seq.*

The phenols are extracted with caustic soda and the soda solution is boiled to remove neutral oils and bases; the phenols are liberated by the addition of hydrochloric acid and their volume is measured.

Method:

The material for test (see under "General" and "Preliminary Treatment of the Sample," opposite) shall be warmed if necessary until completely liquid and transferred to a stoppered separating funnel of suitable capacity. The volume of 10 *per cent w/w* aqueous caustic soda solution required for the test shall be calculated from the content of phenols known to be present

from the determination under Serial No. L.M. 4, page 130, on the assumption that 5 ml of the caustic soda solution are required for each millilitre of crude phenols.

One half the calculated amount of 10 *per cent w/w clear* aqueous caustic soda solution shall be used to rinse the vessel in which the sample was measured and the receiver used in the preliminary distillation if that has been performed. The soda solution shall then be added to the contents of the separating funnel. If the material should contain naphthalene which tends to separate, both it and the caustic soda solution shall be warmed just sufficiently to maintain the naphthalene in solution. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement, the alkaline layer shall be run into a beaker. The upper layer shall be treated again as above, using the remaining half of the calculated amount of soda solution in three equal washes. After settlement, the alkaline layer shall in each case be added to the first soda washing. During the washing processes, the contents of the funnel shall be kept liquid, immersing the funnel in warm water if necessary.

The soda washings shall be boiled vigorously for ten minutes, the initial volume being roughly maintained by the addition of distilled water; fragments of porous or other suitable inert material may be used to prevent bumping. The soda washings shall be cooled to laboratory temperature and if the solution be not clear, it shall be filtered through glass wool previously moistened with saturated aqueous sodium chloride solution; the glass wool shall be washed with 25 ml of saturated sodium chloride solution which shall be added to the filtered soda washings. Methyl orange indicator solution shall be added, followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the mixture shall be kept cool by immersing the container from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a *few* particles undissolved. The whole shall be transferred to a suitable separator and after adequate time for settlement the phenols shall be separated; their volume shall be determined at the temperature at which the material for test was measured and shall be reported as "wet crude phenols."

If a sufficient quantity of crude phenols be obtained, it shall

be examined by the method described in Serial No. C.C. 7, page 216, and the crude phenols less water shall be reported as "dry crude phenols." Alternatively, the crude phenols less water and residue on distillation may be determined according to the method described in Serial No. C.C. 4, page 208, and reported as "distilled dry phenols"; or that test may be carried out in its entirety, whereby the content of phenol will also be obtained.

SERIAL No.: L.M. 5b-38

Pyridine Bases

General:

After suitable pre-treatment of the sample, the pyridine bases are extracted with dilute acid; the extract is neutralized and steam distilled; the pyridine bases are either liberated by the addition of caustic soda or titrated with hydrochloric acid, according to the circumstances.

Material for Test:

The material for test will normally be the phenols-free material from the phenols test, Serial No. L.M. 5a, page 133 (see however under "General" and "Preliminary Treatment of the Sample," page 132).

Method:

The material for test (see above) shall be fractionated in the apparatus and in the manner described in Serial No. C.B. 6b, page 124. The following fractions shall be taken:—(a) up to 195°C (stop-point), the distillate being received in a separating funnel, and the Liebig condenser being used; (b) 195° to 250°C (stop-point), a weighed conical flask being used as the receiver and the Liebig condenser having been replaced by the standard air condenser. Fraction (b) shall be reserved for examination by the method described in Serial No. L.M. 5c, page 137.

Fraction (a) obtained in the separating funnel shall be shaken for two minutes with one fifth of its volume of 25 *per cent w/w* sulphuric acid. The mixture shall be allowed to stand until separation is complete. The acid layer shall be run into a round bottomed flask of suitable size and the distillate washed again with one fifth of its volume of 25 *per cent w/w* sulphuric acid as before. The combined acid washings shall be boiled for ten minutes and after cooling, sufficient 20 *per cent w/w* clear

aqueous caustic soda solution shall be added to neutralize the whole of the acid used. The mixture shall be kept cool during neutralization, to avoid loss of pyridine bases. One hundred millilitres of water shall be added and the mixture distilled in the apparatus used in Serial No. C.C. 5 (Fig. 23, page 213), except that the receiver shall be a flask of suitable size; distillation shall proceed until the drops of condensate shew only a faint opalescence.

The subsequent procedure will depend on whether, assuming sufficient pyridine bases are present, they are to be liberated, or whether, especially if present only in small quantities, only a titration is desired or practicable.

A. LIBERATION OF PYRIDINE BASES

Solid caustic soda shall be added to the aqueous distillate until after thorough agitation the aqueous layer attains a specific gravity of at least 1.35. The mixture shall be allowed to settle and the upper layer, consisting of crude pyridine bases, shall be separated and agitated in a graduated measuring cylinder of suitable size with sufficient solid caustic soda to leave a few particles undissolved. The mixture shall be allowed to stand for at least twelve hours and the volume of the pyridine layer shall be noted at the temperature at which the original sample was measured (see Note).

B. TITRATION OF PYRIDINE BASES

Three drops of a 0.1 *per cent w/v* solution of phenolphthalein in a 50/50 alcohol/water mixture shall be added to the aqueous distillate. If a purple colour should develop, the mixture shall be titrated drop by drop with 0.1N hydrochloric acid until it is just colourless and this liquid—or, if no purple colour should develop on the addition of the phenolphthalein, the liquid after that addition—shall be titrated as follows:—

0.3 ml of a 0.04 *per cent w/v* solution of bromophenol blue (0.04 g of bromophenol blue dissolved in a small quantity of alcohol and the solution diluted to 100 ml with freshly boiled and cooled distilled water) shall be added to the whole of the aqueous distillate (just acid to phenolphthalein) and titration shall be carried out with Normal hydrochloric acid. If only one drop of acid be required, pyridine bases shall be reported as nil. If V be the number of millilitres of Normal hydrochloric acid used in the test, the weight in grams of commercial pyridine bases in the amount of material examined is $0.079 \times V$,

in terms of pyridine whence, assuming a density of 0.982 g/ml, the amount in the sample may be calculated as the percentage by volume.

Note:

Occasions may arise when the amount of total crude bases is required in samples containing a substantial portion distilling above 195°C. In such cases the material described under "Material for Test" shall be washed with a sufficient quantity of 25 per cent w/w sulphuric acid before fractionation. *In these circumstances, the report on the test shall indicate that the total crude bases have been determined.*

SERIAL No.: L.M. 5c-38

Crude Naphthalene

It is to be understood that this determination is only in respect of the naphthalene deposited from solution at 15.5°C.

Method:

Fraction (b) obtained in the preliminary fractionation in Serial No. L.M. 5b, page 135 (see Note) shall be weighed. It shall be warmed if necessary until completely liquid and mixed thoroughly. Fifty grams, or the whole if less than 50 g are available or if the naphthalene content is expected to be low, shall be cooled to $15.5^{\circ}\text{C} \pm 0.5$ Centigrade degree and, after seeding with a small crystal of naphthalene if no crystalline material has appeared by then in the cooled fraction, shall be maintained within those limits of temperature for four hours, with occasional stirring.

The material shall then be transferred to a cold Buchner funnel fitted with a rapid filter paper, and as much oil as possible removed rapidly by a filter pump. The naphthalene shall be placed between sheets of blotting paper and submitted to pressure in a hand screw-press. The oily margins of the cake shall be removed and separately pressed.

The operations, starting with the transfer to the Buchner funnel to the preliminary pressing, shall be conducted with the utmost rapidity.

The pressed naphthalene shall be weighed and its "corrected wet crystallizing-point" shall be determined by the method described in Serial No. C.N. 2, page 248. The crystallizing-point shall be at least 70°C.

The approximate percentage of pure naphthalene in the pressed naphthalene may, if desired, be calculated as—

$$2t - 60$$

where

t = the "corrected wet crystallizing-point" of the pressed naphthalene.

The above formula is, for the present purpose, sufficiently in agreement with that appearing in Serial No. C.N. 3, page 250. The appearance here of the relationship there shewn would give a misleading impression of the value of the results obtained in the present connexion.

Note:

If the sample should distil completely below 195°C in the preliminary fractionation and no fraction (*b*) be available in consequence, naphthalene shall be reported as nil.

SERIAL NO.: L.M. 5d-38

**Loss on Acid-washing; Refined Naphthas, and
Neutral Oils**

Material for Test:

The material for test will normally be the naphtha layer after the final washing with sulphuric acid in the isolation of pyridine bases (Serial No. L.M. 5b, page 135) (see however under "General" and "Preliminary Treatment of the Sample," page 132).

Method:

The material for test (see above) shall be measured at the temperature at which the original sample was measured and transferred to a stoppered separating funnel; 5 *per cent* of its volume of 95 ± 0.5 *per cent w/w* sulphuric acid shall be added. If the proportion of unsaturated hydrocarbons be high, great caution is necessary during this addition and the acid must be introduced in small portions, with vigorous cooling after each addition.

The contents of the separating funnel shall be shaken; care is necessary at first, otherwise the mixture may become uncontrollably hot and burst the vessel or force out the stopper. Any *undue* rise in temperature shall be checked by cooling under running water from time to time. At intervals during the shaking period the pressure may be released by inverting the separating funnel and opening the tap. Shaking shall be

continued for ten minutes and shall be vigorous as soon as the temperature is found to be under control. The mixture shall be allowed to settle for fifteen minutes and 10 to 20 ml of water shall be introduced down the side of the funnel in order to create as little disturbance as possible. Within a minute or two this forms a visible layer between the dark acid and the naphtha, and allows the acid to be removed without risking loss of naphtha. The acid and water layers shall be run off and discarded. A further 10 to 20 ml of water shall be introduced and worked round the sides of the separating funnel to remove any acid tar there deposited. Agitation must be avoided as it may result in the formation of an obstinate emulsion. After settlement, this washing shall also be run off and discarded.

Clear aqueous caustic soda solution (e.g. 10 *per cent* by volume of a 20 *per cent w/w* solution) shall be added and agitated gently with the naphtha, avoiding the formation of an emulsion, until the naphtha changes in colour to yellow or brown. This change may require several minutes and the gentle agitation shall be continued until no further colour change is observed. The mixture shall be allowed to settle until the aqueous portion has separated. The volume of the naphtha layer shall be measured at the temperature at which the original sample was measured.

The loss on acid-washing in millilitres is the difference in volume of the naphtha layer before and after treatment, *plus* the number of millilitres of water recovered during the subsequent fractionation (see below); from this the percentage loss on acid-washing shall be calculated.

The residual naphtha shall be fractionated in the apparatus and in the manner described in Serial No. C.B. 6b, page 124, fractions being taken as shewn below to provide an approximation to the contents of the commercial fractions indicated:—

Up to 100°C	Commercial benzole
100° to 120°C	Commercial toluole
120° to 160°C	Solvent naphtha
160° to 190°C	Heavy naphtha

each temperature being a running-point except 190°C which shall be a stop-point. If the distillation temperature should rise quickly above 100°C after the water has been distilled, the fraction "up to 100°C" will be incorrect. In that event, the water shall be separated from the first fraction in a small separator and the water-free distillate returned to the oil in the

distillation flask after this has been cooled sufficiently, the fractionation being then resumed.

The distillates shall be brought to the temperature at which the original sample was measured and their volumes noted.

If sufficient quantities of the distillates are available, the specific gravities of each may be determined to give some indication of the paraffins content of the respective fractions.

The foregoing fractionation may alternatively be replaced by the Colman and Yeoman test (Serial No. L.B. 27, page 192) in order that the amounts of benzene, toluene, xylenes, etc. in the washed naphtha may be determined.

Statement of Results:

The following shall be reported, the amount of each constituent being calculated as the percentage by volume on the original sample freed from any separated water, except in the case of crude naphthalene which may be reported as indicated:—

PRELIMINARY TESTS

Appearance

Fluidity at laboratory temperature

Specific gravity, $S_{15.5^{\circ}/15.5^{\circ}}$

Water

Distillate at each required temperature, indicating the running-points and stop-points

Temperature at which 95 ml of distillate are collected

Wet crude phenols

COMMERCIAL ANALYSIS

Fractionation:

Colman and Yeoman Test:

Commercial benzole

Benzene

Commercial toluole } or <

Toluene

Solvent naphtha

Xylenes

Heavy naphtha

etc.

Dry crude phenols

Commercial pyridine bases

Neutral creosote, including naphthalene, by difference

Water

Loss on acid-washing

Crude naphthalene: g/ml, or per cent

6. REFINED LOWER BOILING PRODUCTS

Applicability:

The tests described in this section are applicable to samples of refined products consisting essentially of hydrocarbons boiling below 200°C and substantially free from phenols and pyridine bases. In general, such products will have been processed to conform to a commercial specification.

It is intended that heavy naphtha be regarded as falling within the scope of the above.

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least a quart (about 1,200 ml) shall be supplied for a general examination. If the sample is to be examined by a particular set of tests, three times the total amount required for one set of the tests shall be supplied.

(a) GENERAL

SERIAL No.: L.B. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: L.B. 2-38

Colour

Apparatus:

Two standard 50 ml Nessler cylinders (Schedule No. V10, page 363) similar in every respect including colour.

Method:

The sample shall be passed through a filter paper (e.g. Whatman No. 5) about 150 mm in diameter, the first 10 ml of filtrate being rejected. Fifty millilitres of the filtered

sample shall be compared with 50 ml of the solution indicated in the specification which is being followed. The sample and the standard solution, contained in the Nessler cylinders, shall be examined vertically while the cylinders are held three inches above the surface of an opaque opal glass sheet reflecting diffused daylight.

SERIAL No.: L.B. 3-38

Flash-point

Apparatus:

An Abel flash-point apparatus as defined by the Petroleum (Consolidation) Act, 1928.

The apparatus, including the thermometers, shall be certified and marked by the Board of Trade as complying with the requirements of the Act.

Material for Test:

The flash-point is affected by the presence of water; when the test is required on the sample freed from water, the sample shall be dried as follows:—

About 100 g of finely powdered plaster of Paris (calcium sulphate hemihydrate) shall be placed in a dry wide mouthed stoppered glass bottle having a capacity of about 450 ml and an external diameter of from 50 to 60 mm. About 250 ml of the sample shall be introduced and the bottle stoppered. The bottle shall be well shaken for ten minutes and allowed to stand for settling. Sufficient of the dried and settled sample shall be decanted into the testing cup of the Abel flash-point apparatus and the flash-point determined as described below.

Method:

OILS FLASHING BELOW 90°F*

The test apparatus shall be placed for use in a position where it is not exposed to currents of air or draughts.

The heating vessel or water-bath shall be filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the beginning of the test shall be 130 degrees Fahrenheit and no heat shall be applied to the water-bath during the test. When a test

* The usual title has been retained, but this section of the method is applicable only to oils flashing between 66° and 90°F. Oils flashing between 35° and 66°F shall be tested as described in the Note on page 144.

has been completed and it is desired to make another test the water-bath shall be raised again to 130 degrees Fahrenheit, which may conveniently be done while the petroleum cup is being emptied, cooled and refilled with a fresh sample to be tested. The next test is then proceeded with.

If an oil test-lamp is being used it shall be prepared by fitting it with a piece of flat plaited candle-wick, and filling it with colza or rape-oil up to the lower edge of the opening of the spout or wick tube. The lamp shall be trimmed so that when lighted it gives a flame of about 0.15 of an inch diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. A gas test-lamp may be employed, and if so, the size of the jet of flame shall be adjusted to the size laid down above.

The bath having been raised to the proper temperature, the cup shall be placed on a level surface in a good light and the oil to be tested shall be poured into it, until the level of the liquid just reaches the point of the gauge which is fixed in the cup. Before a test is begun the temperature of the oil shall be determined and shall be brought to approximately 60 degrees Fahrenheit. The cover, with the slide closed, shall then be put on to the cup and pressed down so that its edge rests on the rim of the cup, and the cup shall be placed into the bath or heating vessel, every care being taken to avoid wetting the sides of the cup with the oil. The thermometer in the lid of the cup has been adjusted so as to have the correct immersion when the brass collar of the thermometer is properly seated, and its position shall not in any circumstances be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

The test-lamp shall then be placed in position upon the lid of the cup. When the temperature has reached 66 degrees Fahrenheit, the operation of testing shall be begun, the test flame being applied once for every rise of one degree, in the following manner:—

The slide shall be slowly drawn open while a metronome, set so as to beat at the rate of 75 to 80 beats in the minute, beats three times and shall be closed during the fourth beat. A pendulum of 24 inches effective length may be used in place of the metronome, counting one beat from one extremity of the swing to the other.

OILS FLASHING BETWEEN 90° AND 120°F

The air chamber which surrounds the cup shall be filled with cold water to the depth of 1.5 inches and the heating vessel or water-bath filled as usual, but also with cold water. The lamp shall be placed under the apparatus and kept there during the entire operation. The temperature of the oil shall be raised at the rate of 2 to 2.25 Fahrenheit degrees per minute, the testing being carried out as laid down in the previous section, except that the test flame shall first be applied when the temperature has reached 80°F.

The flash-point is the temperature read on the thermometer at the time of that flame application which causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo which sometimes surrounds the test flame during the applications preceding the one which causes the actual flash.

Corrections:

The Petroleum (Consolidation) Act, 1928 does not mention corrections for the effect of barometric pressure on the flash-point. In the absence of specific instructions, the barometric pressure shall consequently be observed and recorded only. If, however, the specification or other instructions which are being followed require the result to be corrected for the effect of barometric pressure, the observed flash-point shall be corrected by adding or subtracting 0.63 Fahrenheit degree for each 10 mm by which the barometric pressure is below or above 760 mm as the case may be.

Note:

The text of the method for "Oils Flashing below 90°F" is precisely that laid down in the Petroleum (Consolidation) Act, 1928 which however makes no provision for liquids having a flash-point below 66°F. The following modifications in the procedure set out for "Oils Flashing Below 90°F" shall be employed for testing liquids flashing between 35° and 66°F:—

OILS FLASHING BETWEEN 35° AND 66°F

The sample shall be cooled to 32°F or below for at least two hours with occasional shaking; the oil cup, thermometer, and cover shall be brought to the same temperature.

The flash-point apparatus shall be prepared as usual, except that the water-bath shall be filled with water at 90°F. The sample, cooled as above described, shall be well shaken and

poured into the cup to the gauge level, the cup then being placed in position without delay. The subsequent procedure shall be exactly as would apply to "Oils Flashing Below 90°F," except that the test flame shall be applied first when the temperature has reached 35°F, and thereafter once for every rise of one degree.

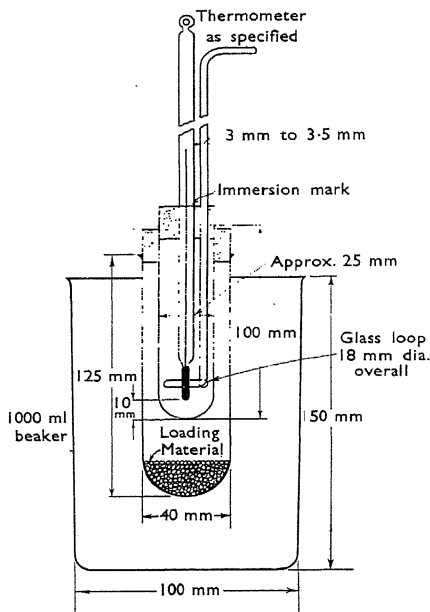
SERIAL No.: L.B. 4-38

Crystallizing-point

Apparatus:

A crystallizing-point apparatus as shown in Fig. 17, below. A glass test tube is placed inside a larger glass test tube which acts as an air jacket. The wider tube is weighted with lead shot or similar material and the inner tube is closed by means of a cork which carries a stirrer and through its centre a standard thermometer (see below). The stirrer is a loop of glass with a glass stem, the loop being arranged to surround the thermometer. The thermometer is so fixed in the cork that the bottom of the bulb is about 10 mm from the bottom of the inner tube. The cork projects for such a distance (about 10 mm) above the top of the inner tube that the immersion mark on the thermometer is level with the top of the cork.

The cooling liquid is contained in a 1,000 ml beaker, about 150 mm in height and 100 mm in diameter at the base. The level of the liquid in the bath shall be at least as high as the level of the sample in the inner tube.



± 5 per cent tolerance on dimensions except where otherwise stated. All diameter dimensions are external.

Fig. 17.—Crystallizing-point—Assembled Apparatus.

A standard thermometer having the range -10° to $+20^{\circ}\text{C}$ (Schedule No. T1a, page 333).

Material for Test:

If the determination be required on the dry sample, and in any event in the case of light oils crystallizing below 0°C , the sample shall be dried before testing by shaking with plaster of Paris (calcium sulphate hemihydrate) and decanting, as described under "Material for Test" in the flash-point test, Serial No. L.B. 3, page 142.

Method:

The inner tube of the apparatus shall be removed from its jacket and a representative portion of about 20 g of the material for test (see above) shall be introduced. A preliminary rapid cooling of the molten mixture shall be carried out to determine the approximate crystallizing-point. The tube shall then be partially immersed in a bath at about five Centigrade degrees above the crystallizing-point, until all but the last traces of crystal are melted. The inner tube shall be replaced in its jacket and the apparatus assembled as shewn in Fig. 17, page 145, with the cooling bath between six and eight Centigrade degrees below the expected crystallizing-point. Thermometer readings shall be taken at intervals of half a minute, with continuous and gentle stirring, this operation being so conducted that seed crystal is present as the temperature of the sample falls to that at which crystallization commences. The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.1 Centigrade degree (but see Note). If supercooling should take place, the constant temperature may be observed immediately after the temperature rise. A temperature rise of one Centigrade degree shall be regarded as the maximum allowable.

If a constant temperature be not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second, and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature rise. The point of intersection shall in this case be reported as the crystallizing-point.

Note:

The crystallizing-point of certain benzoles such as motor benzole, which tend to crystallize with no supercooling and occasionally with some lack of constancy of temperature during the crystallization period, shall be taken as the temperature at which the bulb of the thermometer ceases to be visible.

In such cases, the timing of the thermometer readings may, of course, be omitted.

SERIAL NO.: L.B. 5-38

Neutrality**Method:**

Ten millilitres of distilled water, freshly boiled and cooled in an atmosphere free from carbon dioxide, shall be placed in a clean stoppered 100 ml cylinder, and 0.3 ml of the indicator solution shall be added (see Notes i and ii). Fifty millilitres of the sample shall be added and the stoppered cylinder shaken thoroughly and allowed to settle. The colour of the aqueous layer shall be compared with the colour of the contents of a similar tube prepared with the indicator but without the addition of the sample. The sample shall be reported as acid, alkaline, or neutral as the case may be, *naming the indicator*.

Notes:

(i) It is essential that the cylinders used in the tests be of good quality glass which is quite inert to their contents under the conditions of the test.

(ii) Examples of suitable indicator solutions and their preparation are given below:—

(a) *Methyl orange* (acid—red, neutral—orange, alkaline—yellow): 0.022 g of methyl orange sodium salt shall be dissolved in 100 ml of hot distilled water. The solution shall be cooled and if necessary filtered. It is necessary to add acid to this solution as described below.

(b) *Bromophenol blue* (acid—yellow, neutral—grey and through green to alkaline—bluish violet): 0.04 g of bromophenol blue shall be dissolved in a small quantity of alcohol and the solution diluted to 100 ml with freshly boiled and cooled distilled water. It is necessary to add acid to this solution as described below.

(c) *N.B.A. Mixed indicator* (acid—yellow, neutral—green and through grey and blue to alkaline—violet): 0.04 g of bromocresol green shall be dissolved in 10 ml of alcohol and

0.2 g of alizarin red S shall be dissolved in freshly boiled and cooled distilled water. The two solutions shall be mixed and diluted to 100 ml with freshly boiled and cooled distilled water. This indicator may require the addition of acid or alkali.

Either 0.01N hydrochloric acid or 0.01N caustic soda shall be added to the solution of the indicators as described above, until 0.3 ml of the indicator so treated gives the neutral shade when added to 10 ml of freshly boiled and cooled distilled water.

SERIAL No.: L.B. 6-38

Distillation

Apparatus:

A standard distillation flask (Schedule No. F4, page 365).

Standard thermometers as follows:—

For the distillation of	Thermometer range	Schedule number
Motor benzole, industrial benzole, solvent naphtha and heavy naphtha	50° to 210°C	T9a
Commercial benzoles and com- mercial toluoles	70° to 130°C	T8a
Pure benzole	65° to 90°C	T7a
Pure toluole	105° to 115°C	T11a
Pure xylene and commercial xyloles	130° to 160°C	T12a

The thermometer shall be so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard Liebig condenser with bent end (Schedule No. C1, page 369). The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a clamp to hold the condenser.

A standard 100 ml Crow receiver (Schedule No. V1, page 341).

A standard draught screen (Schedule No. S3, page 373) fitted with an asbestos board having a central hole 40 mm in diameter; the flask shall be placed in position and pressed down to close completely the hole in the board.

The foregoing is illustrated in Fig. 18, opposite, and represents the arrangement shown in B.S. 658—1936, Fig. 1, with the optional items particularized.

A Bunsen burner, the flame of which shall be entirely non-luminous and as small as is conveniently possible.

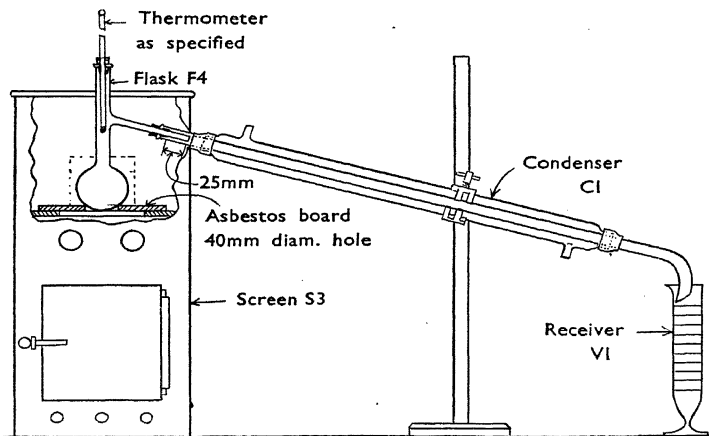


Fig. 18.—Distillation of Refined Lower Boiling Products—Assembled Apparatus.

Method:

One hundred millilitres of the sample shall be measured at laboratory temperature into the 100 ml receiver and transferred to the distillation flask, the contents of the receiver being allowed to drain for fifteen seconds into the flask; a fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping; the flask shall be connected to the condenser and the thermometer inserted. An adequate supply of cooling water shall be passed through the condenser (see Note i). The graduated receiver in which the sample was measured shall be employed without rinsing or drying, to receive the distillate.

The flask shall be heated slowly, especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver; the total period of preliminary heating shall not be less than five nor greater than ten minutes.

The receiver shall be placed so that the condensate will flow down its side; if a determination of the drip-point is to be made, the receiver shall be so placed immediately after

that determination (see Note ii). The distillation shall be continued at the rate of 4 to 5 ml per minute (about two drops per second) and readings of volume and temperature (see under "Temperature Corrections" below) shall be made according to the specification which is being followed.

Should solids tend to deposit during the distillation, the supply of cooling water shall be stopped and even replaced by warm water if necessary, so that such solids are collected in the fraction with which they come over.

The distillate shall be brought to laboratory temperature and measured. The residue in the flask shall be transferred while hot to a graduated receiver, cooled to laboratory temperature and measured. Any difference between the combined volumes of the distillate and residue, and the original volume, shall be recorded as the "distillation loss."

Temperature Corrections:

In the absence of other instructions, the boiling-points shall be corrected for the effect of barometric pressure, according to the instructions on pages 64 to 69.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within:—

(a) ± 0.05 Centigrade degree of the average of the several determinations of the difference in temperature, T Centigrade degrees, at which V_1 ml and V_2 ml of distillate have collected, provided T be not more than 5; and

(b) ± 0.5 ml of the average of the several determinations of the volume of distillate collected at a specified temperature.

Notes:

(i) The temperature and flow of the cooling water in the condenser shall be such that the temperature of the distillate is maintained within ± 5 Centigrade degrees of that at which the sample was measured for test. When this is impracticable, running-point observations cannot be regarded as accurate.

(ii) The temperature reading when 5 *per cent* of the sample has distilled will indicate the presence of low boiling constituents more closely than will the drip-point. Similarly, the temperature reading corresponding to a distillate of 95 *per cent* of the sample is affected by small quantities of high boiling

constituents and serves to detect these better than the dry-point which, under the conditions of the test, is affected by superheating of the vapours.

SERIAL No.: L.B. 7-38

Phenols

Applicability:

The following method is applicable only to samples containing 1 per cent or less of phenols (but see Note).

General:

The phenols are extracted with caustic soda and the soda solution is boiled to remove neutral oils and bases; the phenols are liberated by the addition of hydrochloric acid and their volume is measured.

Method:

Two hundred and fifty millilitres of the sample shall be measured at laboratory temperature into a stoppered separating funnel of about 500 ml capacity. Twenty-five millilitres of a 10 per cent w/w clear aqueous caustic soda solution, measured in the cylinder from which the 250 ml of sample have been emptied, shall be added. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement, the alkaline layer shall be run into a beaker. If the pyridine bases in the sample are to be determined, the upper layer shall be reserved for treatment as described in Serial No. L.B. 8, page 152.

The contents of the beaker shall be boiled vigorously for two minutes; fragments of porous or other suitable inert material may be used to prevent bumping. The contents of the beaker shall be cooled to laboratory temperature and transferred to the phenols flask (Schedule No. V5a, page 347). Methyl orange indicator solution shall be added, followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the contents of the flask shall be kept cool by immersing the flask from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a few particles undissolved; the phenols shall be brought into the graduated portion of the flask by adding saturated sodium chloride solution. After

adequate time for settlement, preferably overnight, the volume of phenols shall be read at the temperature at which the material for test was measured and reported as the percentage by volume of "wet crude phenols."

Note:

The above method may be applied to samples containing more than 1 *per cent* but not more than 3 *per cent* of phenols, by using 100 ml instead of 250 ml of sample, which shall be measured into a stoppered separating funnel of about 250 ml instead of about 500 ml capacity. The amount of caustic soda solution used and the other conditions of test shall remain unaltered.

SERIAL No.: L.B. 8-38

Pyridine Bases

Applicability:

The following method is applicable only to samples containing 1 per cent or less of pyridine bases.

General:

After removal of phenols, the pyridine bases are extracted with dilute acid; the extract is neutralized and steam distilled; the pyridine bases in the distillate are titrated with hydrochloric acid.

Method:

Two hundred and fifty millilitres of the sample shall be treated for the removal of phenols as described in Serial No. L.B. 7, page 151, and the washed sample shall be shaken for two minutes with 20 ml of 25 *per cent w/w* sulphuric acid. The mixture shall be allowed to stand until separation is complete. The lower layer shall be run into a 500 ml round bottomed flask; 100 ml of water and 50 ml of 20 *per cent w/w clear* aqueous caustic soda solution shall be added to the contents of the flask. The mixture shall be kept cool during neutralization, to avoid loss of pyridine bases.

The mixture shall be distilled in the apparatus used in Serial No. C.C. 5 (Fig. 23, page 213) except that the receiver shall be a 250 ml flask; distillation shall proceed until at least 100 ml of distillate have been collected. Three drops of a 0.1 *per cent w/v* solution of phenolphthalein in a 50/50 alcohol/water mixture shall be added to the distillate. If a purple colour should develop, the mixture shall be titrated

drop by drop with 0.1N hydrochloric acid until it is just colourless and this liquid—or, if no purple colour should develop on the addition of the phenolphthalein, the liquid after that addition—shall be titrated as follows:—

0.3 ml of a 0.04 *per cent w/v* solution of bromophenol blue (0.04 g of bromophenol blue dissolved in a small quantity of alcohol and the solution diluted to 100 ml with freshly boiled and cooled distilled water) shall be added to the whole of the aqueous distillate (just acid to phenolphthalein) and titration shall be carried out with Normal hydrochloric acid. If only one drop of acid be required, pyridine bases shall be reported as nil. If V be the number of millilitres of Normal hydrochloric acid used in the test, the percentage (*w/v*) of pyridine bases expressed as pyridine, in the sample is $0.4 \times 0.079 \times V$.

SERIAL No.: L.B. 9-38

Acid Wash Test

General:

Equal volumes of the sample and sulphuric acid of 95 *per cent w/w* strength are shaken together. The colour of the acid layer is compared with a specified standard to give a measure of the degree to which acid refining has been carried.

Colour Standards:

Convenient standards for comparison may be made by dissolving potassium dichromate in a mixture of equal volumes of sulphuric acid (containing 98 parts per 100 by weight of H_2SO_4) and pure distilled water. The standards may be designated by numbers denoting the weight in grams of potassium dichromate in 1,000 ml of the diluted acid, a suitable range being as follows:—

STANDARD DICHROMATE SOLUTIONS

(Weight in grams of potassium dichromate, $K_2Cr_2O_7$, in 1,000 ml of diluted sulphuric acid as above)

0.3
1.0
1.5
2.0
3.0
5.0
7.0
10.0

Method:

The sample shall be passed through a filter paper (e.g. Whatman No. 5) about 150 mm in diameter, the first 10 ml of the filtrate being rejected. Five millilitres of the filtered sample shall be introduced into a dry stoppered colourless glass cylinder about 15 mm in diameter and about 100 mm in height to the shoulder. Five millilitres of 95 *per cent w/w* sulphuric acid (containing 95 parts ± 0.5 per 100 by weight of H_2SO_4) shall be introduced into the tube, which shall be shaken for *exactly* two minutes and allowed to stand undisturbed for *exactly* ten minutes. Comparison shall be made immediately with the specified standard contained in a cylinder identical in all respects, including colour, with the cylinder containing the sample and acid. The colour of the acid layer shall be reported as being equal to a particular colour standard or between two standards.

LOVIBOND COMPARATOR

In order to meet the demand for permanent colour standards the Committee has co-operated with the company The Tintometer Ltd, which is able to supply a Lovibond comparator fitted with a disc containing permanent colour standards corresponding to the dichromate standards described above (see Note). Suitable stoppered shaking tubes are also provided with the apparatus.

Method of Using the Lovibond Comparator:

The sample shall be prepared and shaken for *exactly* two minutes with an equal volume of 95 ± 0.5 *per cent w/w* acid in the special tube provided, as described above. The tube shall be placed in the right-hand compartment of the comparator case and allowed to remain undisturbed for *exactly* ten minutes (the left-hand compartment of the case remaining empty). At the end of this period, the case shall be held about 18 inches from the eye and facing a white light. The disc shall be rotated without delay until the colour in the left-hand window matches or is the next shade darker than that of the sample seen through the right-hand window. The number designating the standard is shewn in the window at the lower right-hand corner of the instrument.

Note on the Lovibond Comparator:

This instrument is lower in price and more convenient than that described in the 1929 edition of "Standard Methods."

Although the use of the latter instrument is no longer regarded by the Committee as obligatory, it should be noted that comparisons made with the previous method and apparatus are equivalent to those obtained by the revised method and the new equipment.

SERIAL No.: L.B. 10-38

Total Potential and Existent Gum

(OXIDATION TEST* FOR STABILITY OF BENZOLE
DURING STORAGE)

Applicability:

The following method is applicable to benzoles distilling not more than 5 per cent at 75°C, not less than 50 per cent at 100°C and not less than 95 per cent at 145°C.

General:

Oxygen is bubbled through the sample while it is heated in a boiling water-bath. Three-quarters of the sample is distilled off, the remainder is evaporated and the residue weighed to give a measure of the total quantity of gum formed by oxidation during the test (potential gum) and any which is present in the sample before the test (existent gum).

Apparatus:

The apparatus shall be as shewn in Fig. 19, facing page 156 (see Note i) and as described below:—

A round bottomed flask of about 250 ml total capacity is attached to a reflux condenser by means of a standard ground joint. A delivery tube for bubbling oxygen through the sample contained in the flask passes down the condenser. The outlet end of this delivery tube is drawn out to 2 mm internal diameter and is just clear of the bottom of the flask. The flask, condenser and oxygen delivery tube are made entirely of an approved resistance glass (see Note ii).

The flask is heated in a water-bath which may conveniently be made of hard rolled copper 22 S.W.G. spun in one piece. The bath is supported on a ring beneath a flat rim on the bath. The bath is provided with a constant level device, a wire stand for supporting the flask and a circular cover. The cover is in two halves, so that when in position it completely

* Adopted by the Technical Committee of the Fifth International Conference of Benzole Producers, September, 1932.

covers the top of the bath with the exception of a hole about 30 mm in diameter, through which the neck of the flask passes. Each half of the cover may conveniently be provided with a handle to facilitate removal. The water-bath is heated by means of a small rose burner.

The oxygen is supplied from a gas cylinder of at least twenty cubic feet capacity and after passing a constant pressure device and a calcium chloride drying tower, is filtered through cotton wool. It is then passed through a capillary type flowmeter (see Note iii) which is connected directly to the delivery tube for bubbling the oxygen through the sample. The flowmeter gauge contains distilled water. If this be coloured with a dye, no volatile acid or alkali must be used to bring the dye into solution. The purpose of the calcium chloride drying tower is to prevent any moisture from condensing in the capillary of the flowmeter.

For the subsequent distillation of the sample and evaporation of the residue, the following apparatus shall also be provided:—

A distillation head of an approved resistance glass, with a standard ground joint to fit the flask (see inset in Fig. 19, opposite).

A 100 ml graduated receiver.

Dishes made in an approved resistance glass, hemispherical in shape (without lip), 66.5 mm ± 2 in external diameter, and 18 g ± 3 in weight.

Material for Test:

It is essential that samples for this test be kept in amber coloured glass bottles fitted with ground glass stoppers. Before filling, the bottle shall be cleaned as specified for the apparatus (see under "Method") and rinsed with the sample. All samples must be kept in the dark. Any sample which does not comply with these requirements shall be reported as unsuitable for test by this method.

Method:

The oxidation process is extremely sensitive to minute traces of impurities; every precaution must therefore be taken to ensure that the whole of the apparatus is clean before starting a test. The reflux condenser shall be connected to the flask and the whole filled with a saturated solution of potassium dichromate in sulphuric acid of about 95 *per cent w/w* strength. After one or two hours the dichromate solution

shall be poured out, the flask and condenser disconnected and both washed thoroughly first with water and then with 5 to 10 *per cent* aqueous caustic soda solution. The whole apparatus shall finally be washed repeatedly with distilled water until all traces of alkali have been removed; this shall be ascertained by testing the washings with litmus paper. The apparatus shall be thoroughly dried by warm air which has been purified by filtration through soda lime and cotton wool.

The oxygen delivery tube and distillation head shall be carefully cleaned in the same way.

The material for test (see above) shall be passed through a filter paper (e.g. Whatman No. 5) about 150 mm in diameter, the first 10 ml of filtrate being rejected. One hundred millilitres of the filtered sample shall be measured into the dry flask which shall be connected to the condenser and set on the wire support in the water-bath. The constant level device shall be adjusted so that the level of the water is the same as that of the sample in the flask. The oxygen delivery tube shall be inserted through the condenser and the inlet connected to the oxygen delivery train.

The cover shall be placed on the water-bath and the flow of water through the condenser started. The water in the bath shall be raised to boiling-point within ten minutes. As soon as the water is boiling, the oxygen shall be turned on and the rate adjusted to $37 \text{ ml} \pm 2$ per minute by means of the tap on the flowmeter. The oxygen shall be bubbled through the benzole for three hours during which the water in the bath shall be kept vigorously boiling, while the temperature of the water passing from the condenser shall not exceed 25°C .

The oxygen shall then be turned off and the flame shall be extinguished. The oxygen delivery tube shall be withdrawn and any moisture wiped from the outside of the condenser. The flask shall be disconnected from the reflux condenser, removed from the water-bath and connected to the distillation head. This in turn shall be attached to an efficient condenser which does not hold up liquid and which is fitted with a bent end. The flask shall be placed on an asbestos sheet, $150 \text{ mm} \times 150 \text{ mm}$, having a central hole 40 mm in diameter. The delivery end of the condenser shall be passed through a cork inserted in the 100 ml graduated receiver. The cork shall also be provided with another tube so that gases and uncondensed vapours evolved during the distillation can be led away from any source of ignition.

The contents of the flask shall be cautiously brought to boiling-point by means of a Bunsen flame, avoiding any sudden evolution of vapour, and distilled at the rate of 4 to 5 ml per minute (about two drops per second) until 75 ml of distillate have collected in the graduated receiver.

The flask shall be disconnected from the distillation head and the contents poured into one of the dishes which has previously been heated in a water or steam oven at between 98° and 105°C, cooled in a dust-free atmosphere for 15 to 20 minutes and weighed to an accuracy of ± 0.2 mg on a balance shielded from direct sunlight. The flask, in an inverted vertical position, shall be allowed to drain into the dish for fifteen seconds. The dish shall be placed at once on a vigorously boiling water-bath where it shall be allowed to remain for $1\frac{1}{2}$ hours, supported in a hole having a diameter of 63 mm ± 1 . The dish shall be protected from dust.

The dish shall be transferred to a water or steam oven where it shall be allowed to remain at a temperature of 98° to 105°C for one hour. It shall be allowed to cool in a dust-free atmosphere for fifteen to twenty minutes and weighed to an accuracy of ± 0.2 mg (see Notes iv and v). The gum content shall be expressed as milligrams of total potential and existent gum per 100 ml of sample.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample giving a result below 15 mg per 100 ml should fall within ± 1 unit of the average of the several readings (see page 4).

Notes:

(i) The apparatus shall not be exposed to direct sunlight during the test.

(ii) The resistance glasses approved for the apparatus used in the test are: Pyrex, Hysil, Monax and Geraete 20.

(iii) The flowmeter shall be such as will give a scale reading of at least 50 mm when passing 37 ml of oxygen per minute at laboratory temperature and pressure; a flowmeter having a capillary 42 mm in length and 0.44 mm in internal diameter will meet this requirement.

The following method of calibrating the flowmeter will be found convenient and provided it is carried out when the

laboratory temperature and pressure are average and steady, is sufficiently accurate for the purposes of the test:—

The scale of the flowmeter is adjusted so that the zero coincides with the level of the liquid in the gauge when no gas is flowing. A 250 ml graduated receiver is filled with water and clamped in an inverted vertical position in a pneumatic trough. Dry oxygen is allowed to pass through the gauge and the reading adjusted to a definite scale reading. After the gauge reading has become constant, the end of the delivery tube, which should be drawn out to about 2 mm diameter, is placed below the mouth of the receiver. As the level of the water passes a prearranged scale reading, a stop watch is started. The flow of oxygen is continued until a mark near the level of the water in the trough is reached (about 200 ml of gas should be passed) and the watch and the oxygen flow are stopped simultaneously. The flowmeter scale reading must have remained constant during the passage of the gas measured.

Then
$$V = (V_2 - V_1) (P - p)$$

and the rate of flow = $\frac{V}{t}$ ml per minute,

where

V = the number of millilitres of gas passed;

V_1 = the number of millilitres of gas in the receiver at the first mark;

V_2 = the number of millilitres of gas in the receiver at the second mark;

P = the barometric pressure in millimetres of mercury;

p = the vapour pressure, in millimetres of mercury, of water at the temperature of the laboratory at the time of calibration; and

t = the time of flow in minutes.

Further measurements are made at different points on the flowmeter scale and the rates of flow are plotted against scale readings. Finally, the gauge is set for the scale reading corresponding to 37 ml per minute, obtained from the mean curve through the results, and a check determination is carried out.

The calibration of the flowmeter shall be checked at least once a month.

(iv) The test must be performed as described above without interruption.

(v) The vapours evolved in this test are *very* explosive, owing to the presence of oxygen, and due precautions must be taken throughout the test.

SERIAL No.: L.B. 11-38

Existent Gum

(RESIDUE ON EVAPORATION)

Method:

One hundred millilitres of the sample shall be measured into a round-bottomed flask as used in the test for potential and existent gum (Serial No. L.B. 10, page 155) and the flask shall be connected to a condenser by means of the ground glass distillation head therein mentioned (see also inset to Fig. 19, facing page 156). Alternatively, a standard distillation flask (Schedule No F4, page 365) made of approved resistance glass may be used (see Note ii to Serial No. L.B. 10). Seventy-five millilitres shall be distilled off at the rate of 4 to 5 ml per minute (about two drops per second) and the residue evaporated in one of the weighed dishes specified in, and in exactly the same manner as described in the test for potential and existent gum.

In the case of fractions distilling less than 50 *per cent* at 100°C and less than 95 *per cent* at 145°C, evaporation on the water-bath shall be continued if necessary for a longer period than 1½ hours and until evaporation is complete.

After the appropriate period on the water-bath, the dish shall be transferred to the water or steam oven where it shall be allowed to remain at a temperature of 98° to 105°C for one hour. It shall be allowed to cool for 15 to 20 minutes in a dust-free atmosphere and weighed to an accuracy of ± 0.2 mg. The gum content shall be expressed as milligrams of existent gum per 100 ml of sample.

SERIAL No.: L.B. 12-38

Unsaturated Hydrocarbons

(BROMINE ABSORPTION)

Reagents:

0.5N Bromide-bromate solution: The bromide-bromate solution shall be made by dissolving 50 g of potassium bromide and 13.92 g of potassium bromate in distilled water, and making up to 1,000 ml.

Approximately 0.2N sodium thiosulphate solution: The sodium thiosulphate solution shall be made by dissolving 49.64 g of sodium thiosulphate in distilled water and making up to 1,000 ml. The solution shall be standardized immediately before use by means of the 0.5N bromide-bromate solution, as follows:—

Five millilitres of the 0.5N bromide-bromate solution shall be run into a ground glass stoppered narrow necked bottle and 10 ml of a freshly prepared 10 *per cent w/v* aqueous solution of potassium iodide shall be added, followed by 20 ml of 10 *per cent w/w* sulphuric acid. The stopper shall immediately be placed in position and the contents of the bottle well shaken.

The liberated iodine shall be titrated with the sodium thiosulphate solution until the contents of the flask become only faintly yellow; starch solution shall then be added and the titration continued until the last traces of blue colour have disappeared.

The factor (n) of the sodium thiosulphate solution is—

$$\frac{v_1}{2v_2}$$

where

v_1 = the number of millilitres of 0.5N bromide-bromate solution used; and

v_2 = the number of millilitres of sodium thiosulphate solution used.

Method:

Twenty millilitres of the sample shall be measured into a 100 ml stoppered narrow necked bottle and 5 ml of 10 *per cent w/v* sulphuric acid shall be added, followed by the 0.5N bromide-bromate solution in sufficient quantity to produce a distinct yellow colour in the sample layer after vigorous shaking for one minute. A reddish-yellow coloration, persisting after the shaking, indicates that too much bromide-bromate solution has been added and in such circumstances the test shall be started afresh.

A further 15 ml of the 10 *per cent w/w* sulphuric acid shall be added in order to complete the liberation of bromine and the bottle shall be well shaken again for one minute. If the contents remain colourless, a further quantity of the bromide-bromate solution shall be added, sufficient to secure a distinct yellow colour.

During the addition of the bromide-bromate solution and the shaking, the action of light must be prevented by wrapping the bottle in which the test is performed, in a dark cloth or in dark paper.

The slight excess of bromine shall be determined by adding 5 ml of a freshly prepared 10 *per cent w/v* aqueous solution of potassium iodide and titrating with the standardized sodium thiosulphate solution, using starch solution as indicator. The back titre shall lie between 0.5 ml and 1.5 ml. If the quantity of thiosulphate solution required should lie outside this range, the test shall be regarded as a trial and a fresh determination shall be made, adjusting appropriately the quantity of bromide-bromate solution added. The amount in grams of bromine absorbed by 100 ml of the sample is—

$$0.40 (0.5 V_1 - nV_2)$$

where

V_1 the number of millilitres of 0.5N bromide-bromate solution used;

V_2 = the number of millilitres of sodium thiosulphate solution used; and

n = the factor for the sodium thiosulphate solution.

SERIAL No.: L.B. 13-38

Non-sulphonable Hydrocarbons

(PARAFFINS AND NAPHTHENES)

General:

The sample is shaken with four times its volume of 99 *per cent* sulphuric acid until sulphonation is complete. The supernatant layer consists essentially of paraffins and naphthenes.

Apparatus:

The sulphonation apparatus described in Schedule No. V7a, page 356, shall be used for samples containing 10 *per cent* or more of non-sulphonable hydrocarbons; that described in Schedule No. V7b, page 356, shall be used for samples containing less than 10 *per cent* (see Note i).

Method:

Eighty millilitres of sulphuric acid (containing 99 ± 0.5 parts per 100 by weight of H_2SO_4 , checked by titration) shall be

poured into the sulphonation apparatus from a graduated cylinder and 20 ml of the sample (see Notes ii and iii) shall be added from a graduated pipette. The stopper shall be inserted, the apparatus inverted and the contents transferred to the stoppered bulb.

The tap shall be opened and the apparatus shaken vigorously for ten minutes, any *undue* rise in temperature being checked by cooling under running water to prevent loss by evaporation. The tap shall be closed and the apparatus placed vertically with the tap downwards until separation has taken place. The volume of unsulphonated hydrocarbons shall be read and the operations of shaking for ten minutes and separation shall be repeated until a substantially constant value is obtained.

The apparatus shall finally be allowed to stand for at least twelve hours and, the lower meniscus of the layer of unsulphonated material having been adjusted to a convenient position on the scale, the volume (V ml) of this layer shall be read at the temperature at which the sample was measured (see Note iv). The percentage by volume of unsulphonated hydrocarbons in the sample is $5V$.

Notes:

(i) A suitable form of spring clip may be used to retain the stopper in position in the sulphonation apparatus during the shaking.

(ii) Samples which yield viscous or solid products with sulphuric acid shall be treated as follows:—

Fifty millilitres or other suitable quantity of the sample shall be shaken in a stoppered vessel (e.g. a separating funnel) with half its volume of about 95 *per cent w/w* sulphuric acid, avoiding any undue rise in temperature, if necessary by adding the acid a little at a time and cooling after each addition. When the reaction is complete and the mixture cool, the mixture shall be added slowly to 200 ml of distilled water, cooling again if necessary. The mixture shall be distilled in the apparatus shewn in Fig. 23, page 213, the separating funnel there shewn being used as the receiver. Porous or other suitable inert material may be added to prevent bumping. When about 100 ml of water have collected, the spirit in the distillate shall be separated from the water and shall be dried by means of a small quantity of 95 *per cent w/w* sulphuric acid. The volume of the dried spirit shall be determined and 20 ml shall be examined by sulphonation as described under "Method"

above. The percentage by volume of unsulphonated hydrocarbons in the sample is then—

$$\frac{5 V_3 V_2}{V_1}$$

where

V_1 = the number of millilitres of sample originally used;

V_2 = the number of millilitres of distilled spirit obtained;
and

V_3 = the number of millilitres of unsulphonated hydrocarbons obtained.

In these circumstances, the report of the test shall indicate that this prior treatment of the sample has been applied.

(iii) Carbon disulphide will be returned as unsulphonated hydrocarbons and if desired this constituent may be removed prior to the test by washing the sample with alcoholic potash as described in the quantitative test for carbon disulphide (Serial No. L.B. 16, page 171), followed by washing with water.

In these circumstances, the report of the foregoing test shall indicate that this prior treatment of the sample has been applied.

(iv) The complete removal of aromatic hydrocarbons may be checked where practicable by applying the formolite reaction as follows:—

About one millilitre of the supernatant layer shall be placed in a test tube; about one millilitre of concentrated sulphuric acid and a few drops of 40 per cent w/w formaldehyde solution shall be added and the mixture shaken.

A dark red precipitate or coloration indicates that aromatic hydrocarbons are present. Since as low a concentration as 0.1 per cent of aromatic hydrocarbons in the supernatant layer will produce a red precipitate, a mere coloration may be ignored. If a positive reaction be given by this test, the sulphonation is incomplete and the test must be repeated using a fresh quantity of the sample.

SERIAL NO.: L.B. 14-38

Total Sulphur

(After G. Claxton, E. G. Hancock and W. H. Hoffert, *Gas J.*, 1932, 199, 94.)

General:

The sample is diluted with alcohol and a known quantity burnt in a lamp, the products of combustion being drawn

diameter. The wick projects about one millimetre above the top of the glass tubing. Round the burner is fitted a short sleeve of silica tubing which rests on a copper spiral, the size of the flame being controlled by the height of the sleeve.

The burner is attached by means of a capillary tube and a three-way tap to a 50 ml Class A or Class B burette reading to 0.1 ml. A small cup containing mercury for sealing the chimney mounting (see below) is sealed to the capillary tube below the burner. The three-way tap is situated at the lowest point of the capillary tubing and has an outlet for draining the burette. The tap shall be reasonably free from leakage (see Note i). A side tube, terminating at the axis of the burette in a fine jet about 0.5 mm in diameter, is sealed at right-angles through the side of the burette below the graduations. A short piece of rubber tubing carrying a screw clip is attached to the side tube.

The burner and the burette must both be vertical when in operation and the height of the jet must be 16 mm \pm 1 below that of the top of the burner. It is important when constructing the lamp to ensure that this dimension is within the limits indicated.

The principle of operation of the lamp is as follows: The top of the burette is closed with a cork during use and air can then only enter through the jet as liquid from the burette burns. This point is thus maintained at atmospheric pressure and hence the level of the liquid in the burner remains constant.

(b) *Chimney Mounting and Chimney*: A ram-leather is cemented to a short glass cylinder tapering at the lower end so that it fits into the small cup containing mercury and attached to the lamp below the burner. The ram-leather contains mercury to form a seal for the base of a chimney made of heat resisting glass. A side tube is fitted at the lower end of the glass cylinder and is attached to two air scrubbers in series. The body of the chimney mounting is loosely packed with glass wool to prevent air currents which would distort the flame and cause it to smoke.

The first air scrubber consists of an absorption tower, not less than 50 mm in diameter and 120 mm in effective height, filled with sifted broken glass moistened with dilute sulphuric acid; the second scrubber is a similar tower filled with sifted broken glass moistened with 20 per cent w/w aqueous caustic soda solution. The filling in these scrubbers must be such as

to cause the minimum back pressure. The connecting tubes should be about 10 mm in diameter.

The chimney is drawn out to a bend, the outlet of which slips over the central tube of the glass cup forming the uppermost part of a combined wash flask and scrubber (see below).

(c) *Wash Flask and Scrubber*: The inlet tube to the wash flask terminates in a bulb perforated with at least eight holes about half a millimetre in diameter and of equal size. The inlet tube carries round it a scrubber filled with finely graded broken glass (passing an 8-mesh sieve but retained on a 10-mesh sieve). The scrubber makes a gas tight ground joint with the neck of the flask, which is of the usual flat bottomed type with a wide neck, preferably somewhat shortened. The inlet tube and the flask must both be vertical. The outlet tube of the scrubber is connected to a suitable suction pump capable of drawing through the apparatus sufficient air to maintain smokeless combustion.

It is essential that the wash flask and the scrubber be of good quality glass which does not cause the hydrogen peroxide solution therein (see below) to become alkaline. Similarly, the glass packing in the scrubber must be inert to the hydrogen peroxide solution.

The apparatus is suitably mounted on a wooden stand, the chimney mounting and wash flask being supported on a bracket and a slot being cut in the bracket to take the chimney mounting and ram-leather. The lamp shall be placed so that it is not exposed to direct sunlight or to draughts.

Reagent:

About 100 ml of approximately 1.5 *per cent w/w* hydrogen peroxide solution shall be made by suitably diluting concentrated hydrogen peroxide with distilled water (see Note ii). Six to ten drops of a 0.04 *per cent w/v* solution of bromophenol blue shall be added and the solution neutralized by the cautious addition of 0.1N sodium carbonate solution, using Normal sodium carbonate solution in the early stages if necessary. The colour change of bromophenol blue is from yellow (acid) through grey and green to bluish violet (alkaline). The end-point shall be determined by matching the colour against a standard made by the addition of the same quantity of the indicator to 100 ml of distilled water and adding 0.1N hydrochloric acid or sulphuric acid to bring the colour of the solution to the intermediate grey shade.

Method:

A portion of the neutralized hydrogen peroxide solution prepared as described under "Reagent" shall be used to fill the lute round the inlet tube of the wash flask and the remainder of the solution shall be poured down the scrubber into the flask, where it must cover the whole of the perforated bulb.

The asbestos wick and silica sleeve of the burner shall be cleaned by igniting them in a blow-pipe flame before each determination. When an existing wick becomes frayed, a new wick treated in this manner shall be provided.

The stock solution for burning shall be prepared by measuring 20 ml of the sample into a 100 ml graduated flask which is about two-thirds full of "blending methanol" or ethyl alcohol (see Note iii). The solution shall be made up to the graduation mark with more of the alcohol, the whole being thoroughly mixed by shaking. A few millilitres of the stock solution shall be used to rinse the burette and the burner. With the lamp in position and the tap to the burner and the side tube to the jet closed, sufficient of the stock solution shall be poured into the burette to fill it to about the zero mark. The top of the burette shall immediately be closed with a well-fitting cork. The tap shall be opened to the outlet at the bottom of the lamp, when a small quantity of the solution will run out. (If the liquid continues to flow from the burette, this shews that air is entering at the top of the burette and that the cork requires attention.)

The tap shall be turned so that the burner is connected to the burette and the side tube to the jet shall be opened. The solution in the burette will flow into the burner to a height governed by the height of the jet. The wick shall be placed in the burner, care being taken that no air bubbles are trapped. The copper spiral and silica sleeve shall be placed in position on the burner and the lamp shall be lit, warming the silica sleeve if necessary. The size of the flame shall be adjusted by altering the height of the silica sleeve (*not* by adjusting the wick) until the flame is about half the maximum size that gives smokeless combustion. The lamp shall then be allowed to burn in the open until bubbles begin to rise from the jet at regular intervals.

The suction on the outlet tube of the wash flask shall be started, the chimney placed in position, and the burette reading immediately noted. Care must be taken to draw sufficient air through the apparatus to ensure smokeless

combustion. If, in order to obtain sufficient air, it be necessary to reduce the pressure in the chimney by more than 7.5 mm of water below atmospheric pressure, the resistance to the flow shall be reduced by altering the packing in the air scrubbers. The reduction in pressure is conveniently indicated by the liquid in the seal which connects the chimney with the scrubber.

After at least 10 ml of the stock solution have been burnt (see Note iv), the burette shall be read and the chimney immediately removed. The wash flask shall be disconnected from the suction pump, and the scrubber, the inlet tube and the narrow portion of the chimney rinsed into the flask with distilled water (see Note v). The hydrogen peroxide solution in the cup at the top of the wash flask shall also be washed into the flask with distilled water.

A further six to ten drops of a 0.04 *per cent w/v* solution of bromophenol blue shall be added and the contents of the flask shall be titrated with 0.1N sodium carbonate solution. The end-point shall be determined by matching the colour of the solution against the colour standard prepared for matching the neutralized hydrogen peroxide solution (see above, under "Reagent").

A blank test shall be carried out on the alcohol by burning 40 to 50 ml in the lamp.

The percentage by weight of sulphur in the sample is—

$$\frac{0.802 \times (V - v)}{\rho \times M}$$

where

V = the number of millilitres of 0.1N sodium carbonate solution used in the test;

v = the number of millilitres of 0.1N sodium carbonate solution which, calculated from the blank test on the alcohol, would be required if an amount of alcohol equal to 4M/5 were burnt in the lamp;

ρ = the density in grams per millilitre of the sample at the temperature of test; and

M = the number of millilitres of stock solution of the sample burnt in the lamp.

When the temperature of test does not differ by more than ± 5 Centigrade degrees from 15.5°C, it is sufficiently accurate to use in the calculation either the density or the specific gravity of the sample at 15.5°C.

If desired, a further determination may be carried out by refilling the wash flask with fresh neutralized hydrogen peroxide solution and burning at least another 10 ml of the stock solution.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 0.01 unit of the average of the several results (see page 4).

Notes:

(i) The three-way tap shall be tested for leakage as follows:—

The key shall be freed from all traces of grease, dipped into water and replaced in the barrel. Both the side tube and the burner shall be closed by any suitable means. The burner and the burette shall be filled with water and the outflow from the tap shall also be filled with water. No cork shall be inserted in the top of the burette. The water level in the burette shall be 700 mm above the lowest portion of the outflow of the tap, which shall be turned to connect the burette and the burner. After standing for three hours, the fall in level of the water in the burette shall not exceed 0.72 ml.

(ii) Hydrogen peroxide is usually purchased in 10-, 20-, 40- or 100-volumes strengths, corresponding respectively to 3, 6, 12 or 30 *per cent w/w* by weight of hydrogen peroxide in solution.

(iii) "Blending methanol" has the advantage that it enables a larger flame to be used; made synthetically, it is usually almost anhydrous. Whichever alcohol be used, it must be at least 90 *per cent v/v* in strength.

(iv) The method is applicable to a considerable range of products. With samples of low sulphur content, sufficient of the liquid in the burette shall be burnt to give a reading of at least 1 ml (preferably more) when the contents of the wash flask are titrated with 0.1N sodium carbonate solution. If the sample be found to contain more than 2 *per cent* of sulphur, a further test shall be carried out, increasing the dilution of the original sample with alcohol.

(v) The distilled water used for washing down the scrubber shall have previously been brought to the same end-point as the neutralized hydrogen peroxide solution, by the addition of six to ten drops of a 0.04 *per cent w/v* solution of bromophenol blue to each 100 ml of the distilled water, followed by 0.1N

hydrochloric or sulphuric acid until the colour standard is matched.

SERIAL No.: L.B. 15-38

Carbon Disulphide

(QUALITATIVE TEST)

General:

The sample is shaken with phenylhydrazine when, if carbon disulphide be present, a white precipitate is formed.

Method:

Five millilitres of the sample shall be shaken for five minutes with one millilitre of freshly distilled phenylhydrazine in a 10 ml stoppered graduated cylinder. Observation of the contents of the cylinder shall be made after allowing the cylinder and contents to stand for half an hour. A white precipitate indicates the presence of carbon disulphide.

Note:

As the quantity of precipitate is roughly proportionate to the quantity of carbon disulphide in the sample, the test may be calibrated for use as a roughly quantitative method by noting the volumes of precipitate produced from samples containing known quantities of carbon disulphide. The method described in Serial No. L.B. 16, below, shall be used if the *exact* amount of carbon disulphide present is to be reported.

SERIAL No.: L.B. 16-38

Carbon Disulphide

(QUANTITATIVE TEST)

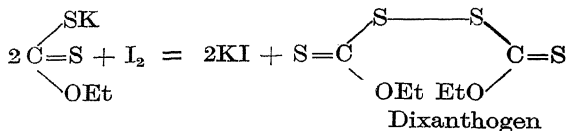
Applicability:

The procedure described under "Method" below is applicable to samples containing 0.1 to 2.0 per cent of sulphur present as carbon disulphide (but see Note).

General:

The sample is treated with alcoholic potash, the resulting potassium xanthate is extracted together with the excess of caustic potash and after acidification of the washings the solution thus obtained is titrated with standard iodine solution.

The reaction between iodine and the xanthate is represented thus:-



Reagents:

Approximately Normal alcoholic caustic potash solution: Thirty grams of caustic potash shall be dissolved in 20 ml of water and the resulting solution added to 500 ml of industrial methylated spirit, 74°O.P. The mixture shall be well shaken until homogeneous and allowed to stand for twenty-four hours. During this time any potassium carbonate which may have been present in the caustic potash will separate out. The solution shall be filtered through glass wool.

Alternatively, for use in the testing of many products, "blending methanol" may replace the methylated spirit, in which case the 20 ml of water may be omitted as unnecessary for bringing the caustic potash easily into solution.

0.1N Iodine solution.

Starch solution, freshly prepared in the manner usual for iodine titrations.

Material for Test:

Hydrogen sulphide and sulphur dioxide affect the determination and if present in more than traces, must be removed by shaking the sample with 5 per cent by volume of 5 per cent w/w clear aqueous caustic soda solution. Similarly mercaptans, which also affect the test, must be removed by shaking with 20 per cent by volume of one per cent w/v aqueous silver nitrate solution. These treatments must be followed by a wash with water.

Method:

Twenty millilitres of the material for test (see above and Note) shall be mixed with about 10 ml of the alcoholic potash solution in a short-stemmed stoppered separating funnel and the mixture, which shall be homogeneous, shall be allowed to stand for at least five minutes. The mixture shall then be extracted three times, each time with 20 to 25 ml of water.

The combined aqueous extracts shall be acidified by the addition of about 7 ml of approximately 2N acetic acid and titrated with the standard iodine solution, using the starch solution as indicator in the usual manner. When the titration is complete, the starch iodide colour fades slowly, due to further oxidizing action of the excess iodine. The distinction between the instantaneous oxidation of the xanthate and this slow reaction is however well marked and there is no difficulty in determining the required end-point.

The percentage by weight of sulphur present as carbon disulphide in the sample is:—

$$\frac{0.64v}{V\rho}$$

where

V the number of millilitres of sample used;

v = the number of millilitres of 0.1N iodine solution used;
and

ρ = the density of the sample at the temperature of the test.

When the temperature of test does not differ by more than ± 5 Centigrade degrees from 15.5°C, it is sufficiently accurate to use in the calculation either the density or the specific gravity of the sample at 15.5°C.

Note:

The quantities of sample, alcoholic potash, water for extraction and acetic acid are suitable for samples containing 0.1 to 2.0 *per cent* of sulphur present as carbon disulphide. When samples containing 2.0 to 4.0 *per cent* of sulphur present as carbon disulphide are being examined, the quantities of alcoholic potash, extraction water and acetic acid shall be doubled. When determining the carbon disulphide in fore-runnings containing over 4 *per cent* of sulphur as carbon disulphide, 10 ml of the alcoholic caustic potash shall be used for each 2 *per cent* of sulphur present as carbon disulphide and each water wash shall be with a volume twice that of the alcoholic caustic potash used. An aliquot portion of the aqueous extract shall be taken in order to avoid excessively high iodine titres. When the sample contains less than 0.1 *per cent* of sulphur as carbon disulphide, the quantity of sample shall be increased to 50 ml or a greater quantity if desired.

Hydrogen Sulphide and Mercaptans**(QUALITATIVE TEST)****Reagents:**

Sodium plumbite solution: The sodium plumbite solution shall be prepared by dissolving about 125 g of sodium hydroxide in one litre of distilled water; 60 g of litharge (PbO) passing a 100-mesh sieve shall be added to the solution and the mixture boiled for half an hour. It shall then be allowed to settle and the clear liquid decanted or siphoned; if the liquid be not clear, it shall be filtered through a mat of asbestos.

The sodium plumbite solution may alternatively be prepared by adding a filtered solution of 25 g of lead acetate crystals in 200 ml of water to a solution of 60 g of sodium hydroxide in 100 ml of water, the combined aqueous solutions being heated on a steam bath for thirty minutes and then diluted to one litre.

The sodium plumbite solution shall be kept in a tightly corked bottle and shall be filtered before use if not clear.

Sulphur: Dry flowers of sulphur, B.P.

Material for Test:

If it be desired to apply this as a test for mercaptans only, hydrogen sulphide shall first be removed by shaking the sample vigorously in a stoppered separating funnel with 5 per cent of its volume of an aqueous solution containing 10 g of cadmium chloride per 100 ml, to which 1 ml of concentrated hydrochloric acid has been added. After settlement and separation, the treated sample shall be washed with water. After further settlement, 10 ml of the supernatant layer shall be withdrawn by means of a pipette.

Method:

Ten millilitres of the material for test (see above) and 5 ml of the sodium plumbite solution shall be shaken vigorously together for 15 seconds in a glass stoppered cylinder of 50 ml capacity and about 25 mm in internal diameter.

Opalescence followed by darkening in colour at this stage, i.e. before adding the sulphur, indicates the presence of both mercaptans and elementary sulphur.

If hydrogen sulphide was present in the sample and not removed, there will be an immediate black precipitate.

Hydrogen sulphide being absent, a small quantity of the sulphur (0.05 to 0.1 g), such that practically all of it floats on the interface between the sample and the sodium plumbite solution, shall be added. The cylinder and contents shall be shaken for fifteen seconds and allowed to settle for one minute, when observation shall be made of the contents of the cylinder (see Notes i and ii).

Interpretation of Results:

If the sample and the sodium plumbite solution shew no change of colour and the sulphur film remains bright yellow, the test shall be reported as "Negative." If the sample or the sodium plumbite solution be discoloured, or the yellow colour of the sulphur at all masked, the test shall be reported as "Positive." If the discoloration be very slight, the test may be reported as "Slightly positive."

Notes:

(i) Oxidized samples may contain peroxides giving a brown precipitate not darkened by the addition of the sulphur; this must not be confused with the effect of mercaptans. The presence or absence of peroxides may be ascertained by shaking the sample with 25 per cent by volume of a freshly prepared 5 per cent *w/v* solution of potassium iodide to which a few drops of starch solution have been added. Peroxides will immediately produce a strong blue coloration.

(ii) Carbon disulphide, particularly in concentrations exceeding 0.4 per cent of sulphur present as carbon disulphide, causes a darkening of the aqueous layer on standing. The test is therefore unreliable for samples containing high amounts of carbon disulphide and caution must always be exercised not to confuse this darkening with the immediate blackening due to hydrogen sulphide or that produced by mercaptans. Similarly, certain phenolic substances (which may have been introduced as inhibitors) cause coloration of the aqueous layer and if their presence be expected, a blank test with caustic soda in place of the sodium plumbite solution must first be carried out for the purpose of comparison.

(iii) The above method is, apart from editorial unification with the present publication, substantially the same as the method Serial Designation G. 33 in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 82.

Mercaptans**(QUANTITATIVE TEST)**

(After P. Borgstrom and E. E. Reid, *Industr. Engng Chem., Anal. Edn.*, 1929, 1, 186.)

General:

The mercaptans are precipitated as silver mercaptides by silver nitrate and the excess of silver nitrate is determined by titration with ammonium thiocyanate.

Reagents:

0.05N *Silver nitrate solution.*

0.05N *Ammonium thiocyanate solution.*

Five per cent w/v aqueous ferric alum solution, to which sufficient nitric acid has been added to cause the disappearance of the brown colour.

Material for Test:

If the colour of the sample would interfere with the test, the sample shall be submitted to distillation prior to examination.

If it be desired to determine mercaptans in samples containing hydrogen sulphide, the latter shall first be removed by shaking the sample vigorously in a stoppered separating funnel with 5 per cent of its volume of an aqueous solution containing 10 g of cadmium chloride per 100 ml, to which 1 ml of concentrated hydrochloric acid has been added. After settlement and separation, the treated sample shall be washed with water. After further settlement, 20 ml of the supernatant layer shall be withdrawn by means of a pipette.

Method:

Twenty millilitres of the material for test (see above) shall be measured into a 100 ml bottle provided with a tightly fitting glass stopper, and 2 ml of the ferric alum solution shall be added. The 0.05N silver nitrate solution shall be added from a burette until after two minutes *vigorous* shaking the addition of four drops of the ammonium thiocyanate solution, also from a burette, does not produce a permanent reddish tinge in the aqueous layer; about 12 ml of the silver nitrate solution will be required for each 0.1 per cent of sulphur present as mercaptans. Sufficient of the ammonium thiocyanate solution shall be added

to produce a permanent red colour after two minutes *vigorous* shaking. More of the silver nitrate solution shall be added with *vigorous* shaking and in sufficient quantity to destroy the red colour. The addition of the ammonium thiocyanate solution shall be continued drop by drop and with intermittent *vigorous* shaking until the red coloration just reappears. If difficulty be found in the final titration because of the formation of emulsions, one or more additions of 10 ml of ethyl alcohol or methanol may be made in order to break the emulsion.

The percentage by weight of sulphur present as mercaptans in the sample is:—

$$\frac{0.00802 \times (V_1 - V_2)}{\rho}$$

where

V_1 = the number of millilitres of 0.05N silver nitrate solution used;

V_2 = the number of millilitres of 0.05N ammonium thiocyanate solution used; and

ρ = the density of the sample at the temperature of the test.

When the temperature of test does not differ by more than ± 5 Centigrade degrees from 15.5°C, it is sufficiently accurate to use in the calculation either the density or the specific gravity of the sample at 15.5°C.

SERIAL No.: L.B. 19-38

Corrosive Sulphur

(COPPER STRIP TEST)

Apparatus:

A thick walled test tube, about 40 mm in diameter and about 175 mm in length, fitted by means of a cork to a reflux condenser having a water jacket about 160 mm in length.

A piece of electrolytic copper foil 30 mm \times 40 mm.

Method:

The copper foil shall be cleaned on both sides until the surface is highly polished, by rubbing it with a cork moistened with distilled water and dipped in dry emery powder of the finest grade (passing a 180/200-mesh sieve); the foil shall be wiped with filter paper moistened with pure acetone, care being taken that it is not touched by the fingers. The cleaned foil shall be cut

into two strips each 30 mm \times 20 mm, and one strip shall be placed in a clean test tube and the tube corked. The other shall be introduced into the thick walled test tube, containing 50 ml of the sample under examination; this test tube shall be fitted to the reflux condenser and allowed to remain for two hours in a water-bath maintained at $50^{\circ}\text{C} \pm 2^{\circ}$.

The appearance of the copper strips shall then be compared, the result of exposure to the sample being reported under one or other of the following descriptions:—

- (1) No change.
- (2) Slight discoloration.
- (3) Brown or peacock shade.
- (4) Steel grey.
- (5) Black, not scaled.
- (6) Black scale.

Notes:

(i) Both free sulphur and hydrogen sulphide discolour the copper under the conditions of the test.

(ii) The extent to which corrosion takes place may be affected by the presence of various substances, including peroxides, which will inhibit the corrosion. The presence or absence of peroxides may be ascertained by shaking the sample with 25 per cent by volume of a freshly prepared 5 per cent *w/v* solution of potassium iodide to which a few drops of starch solution have been added. Peroxides will immediately produce a strong blue coloration.

SERIAL No.: L.B. 20-38

Corrosive Sulphur

(Gravimetric Test)

(After F. H. Garner and E. B. Evans, *J. Instn. Petrol. Tech.* 1931, 17, 451.)

General:

The sample is refluxed with "copper-bronze" powder; the resulting sulphide is oxidized to sulphate which is precipitated as barium sulphate and weighed.

Method:

The determination shall be carried out in an atmosphere free from sulphur compounds.

Half a gram (within 0.01 g) of "copper-bronze" powder shall

be placed in a 250 ml flask fitted with a reflux condenser to which may be fixed a lead-off tube for uncondensed vapours. One hundred millilitres of the sample shall be measured into the flask and boiled for one hour. During the boiling period, the contents of the flask shall be shaken round occasionally. The contents of the flask shall be allowed to cool somewhat and the copper-bronze powder to settle, and the sample shall be carefully decanted through a filter paper (e.g. Whatman No. 40).

The copper-bronze shall be washed with 50 ml of petroleum spirit or pure benzole (previously freed from free sulphur by boiling with copper-bronze powder) and this also decanted through the filter. The filter paper and flask shall be placed in a steam oven for a few minutes to dry. Fifty millilitres of distilled water shall be placed in the flask and an excess of bromine (1 ml) added until all the bronze and sulphide have dissolved. Three millilitres of concentrated hydrochloric acid shall be added, the solution washed into a 400 ml beaker and the excess of bromine boiled off. The filter paper shall also be similarly treated with bromine in a separate beaker and the resulting liquid added to the main bulk.

The bromine-free solution shall be filtered and the filtrate reduced to about 75 ml by boiling. Ten millilitres of hot 10 per cent *w/v* aqueous barium chloride solution shall be added in a fine stream or by drops to the hot solution, the contents of the beaker being stirred during this addition and for two minutes thereafter. The solution shall be allowed to stand overnight or shall be kept hot for one hour on the steam bath or hot plate and the precipitate allowed to settle for one hour. The supernatant liquid shall be filtered through an ashless quantitative filter paper (e.g. Whatman No. 44) and the precipitate washed with distilled water, first by decantation and then on the filter, until free from chloride. The paper and precipitate shall be ignited in the usual manner and weighed.

A blank experiment shall be carried out in order to allow for traces of sulphur in the copper-bronze, bromine and other reagents.

The corrected weight of barium sulphate in milligrams, when multiplied by 0.1375, gives the number of milligrams of corrosive sulphur per 100 ml of sample.

Notes:

- (i) The extent to which the reaction with the copper-bronze

proceeds may be affected by the presence of various substances, including peroxides, which have an inhibiting effect. The presence or absence of peroxides may be ascertained by shaking the sample with 25 *per cent* by volume of a freshly prepared 5 *per cent w/v* solution of potassium iodide to which a few drops of starch solution have been added. Peroxides will immediately produce a strong coloration.

(ii) The above method is, apart from editorial unification with the present publication, identical with the method Serial Designation G4b in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 151.

SERIAL No.: L.B. 21-38

Thiophen

(Qualitative Test)

Applicability:

The following method is only applicable when the sample is completely free from unsaturated hydrocarbons (see Note).

Method:

One millilitre of the sample (see Note) shall be added to 25 ml of a 0.05 *per cent w/v* solution of isatin in pure sulphuric acid in a 100 ml cylinder; 25 ml of pure sulphuric acid, containing one drop of concentrated nitric acid, shall be added and the contents of the cylinder mixed. A blue colour indicates the presence of thiophen.

Note:

The absence of unsaturated hydrocarbons from the sample may be ascertained by examining a portion as follows:—

Ten millilitres of Normal sulphuric acid shall be placed in a small stoppered cylinder together with one millilitre of 0.5N bromide-bromate solution. Two millilitres of the washed spirit shall be added and the whole shaken vigorously for one minute.

If the colour be discharged, the presence of unsaturated hydrocarbons is indicated. The sample may not however be subjected to acid-washing and then examined as under "Method" above, as such washing would inevitably result in the removal of some at least of the thiophen. There is no known reliable method for the detection of thiophen in the presence of unsaturated hydrocarbons.

SERIAL NO.: L.B. 22-38

Thiophen

(Quantitative Test)

(After P. E. Spielmann and S. P. Schotz, *J. Soc. chem. Ind.*, 1919, **38**, 188r.)

Applicability:

The following method only gives accurate results when the sample is completely free from unsaturated hydrocarbons (see Note).

General:

The sample is shaken with basic mercury sulphate solution which precipitates an additive compound with thiophen.

Apparatus:

A glass-stoppered test tube, about 150 mm in length and about 22 mm in diameter.

A Gooch crucible, prepared with acid treated asbestos and a well fitting perforated porcelain plate on the asbestos. Alternatively, a Jena glass filtering crucible No. 2 G3 may be used. In either event the crucible shall be dried before use in an oven at 110° to 115°C, cooled and weighed.

Reagent:

The reagent shall be prepared by placing about 5 g of mercuric oxide in a porcelain basin, adding 100 ml of distilled water and then 20 ml of concentrated sulphuric acid with vigorous stirring. The solution, which should be colourless, shall be filtered and stored in a glass-stoppered bottle.

Method:

About 20 ml of the reagent shall be measured into the stoppered test tube, followed by 2 ml of the sample from a burette (see Note). The stopper, wetted with distilled water, shall be inserted quickly into the test tube and the latter fixed in a shaking machine the speed of which shall be adjusted so that the liquid in the tube is vigorously agitated. After three hours' shaking the precipitate shall be washed completely into the crucible by means of hot water, care being taken to ensure that no precipitate is lost by ebullition of the sample.

If any particles of the precipitate pass into the filtrate, the latter must be refiltered, but emulsified sample must not be mistaken for the finely divided solid. The precipitate shall be washed with hot water until the filtrate is neutral to litmus paper, and the crucible and contents dried in an oven at 110° to 115°C and weighed.

The composition of the precipitate agrees quantitatively with the formula $(\text{HgO}, \text{HgSO}_4)_2\text{C}_4\text{H}_4\text{S}$; the percentage by weight of sulphur present as thiophen in the sample is therefore—

$$\frac{0.029 \times 100W}{2\rho}$$

where

W = the weight of precipitate in grams; and

ρ = the density of the sample at the temperature of the test.

When the temperature of test does not differ by more than ± 5 Centigrade degrees from 15.5°C, it is sufficiently accurate to use in the calculation either the density or the specific gravity of the sample at 15.5°C.

Limits of Accuracy:

The results obtained should fall within ± 0.005 unit of the true value (see page 4).

Note:

The absence of unsaturated hydrocarbons from the sample may be ascertained by examining a portion as follows:—

Ten millilitres of Normal sulphuric acid shall be placed in a small stoppered cylinder together with two millilitres of the washed spirit; one millilitre of 0.5N bromide-bromate solution shall be added and the whole shaken vigorously for one minute.

If the colour be discharged, the presence of unsaturated hydrocarbons is indicated. The sample may not however be subjected to acid-washing and then examined as under "Method" above, as such washing would inevitably result in the removal of some at least of the thiophen. There is no known reliable method for the determination of thiophen in the presence of unsaturated hydrocarbons.

(b) SPECIAL FRACTIONS

SERIAL No.: L.B. 23-38

Benzene, Toluene and Xylene in Certain Mixtures**General:**

The following methods (Serial Nos L.B. 23a and L.B. 23b) and those described in Serial Nos L.B. 24, page 186, and L.B. 25, page 188, are strictly applicable to a limited number of mixtures containing benzene, toluene and xylene only, but are employed in cases of refined materials, i.e. acid-washed materials, for example 90's benzole, 90's toluole, refined toluole, etc. In these cases, any assumption as to the distribution of impurities, e.g. paraffins, is entirely arbitrary unless confirmed in some other manner.

The Committee hopes that at some later date it will prove possible to correlate all these tests and thus to analyse any mixture of benzene, toluene and xylene by one simple distillation, but up to the present there is no sign that this can be done. In the meantime, analysts must decide for themselves which of the tests is applicable in any particular case. The applicability of each test is indicated.

SERIAL No.: L.B. 23a-38

Benzene and Toluene in Certain Mixtures

(After H. G. Colman and E. W. Yeoman, *J. Soc. chem. Ind.*, 1919, 38, 57r.)

Applicability:

The following method is strictly applicable only to mixtures containing 55 to 97 per cent of benzene, the remainder being toluene.

Mixtures of benzene and toluene outside this range may be examined after the addition of an appropriate known amount of pure benzene or pure toluene as required.

The method is useful in the examination of 90's benzole.

Apparatus:

A distillation apparatus as described in Serial No. L.B. 6, page 148, except that a standard distillation flask (Schedule No. F2, page 365) shall replace that therein specified (Schedule

No. F4); the flask shall be fitted with a standard thermometer having the range 70° to 130°C (Schedule No. T8a, page 333).

A standard 100 ml Crow receiver (Schedule No. V1, page 341).

Method:

One hundred millilitres of the sample, measured at laboratory temperature, shall be distilled at the rate of 7 ml per minute (about three drops per second). When the temperature reaches 85°C (see under "Temperature Corrections" below) the flame shall be extinguished, the condenser allowed to drain completely and the volume of the distillate measured at laboratory temperature. If the sample should contain more than 20 *per cent* of toluene, however, the distillation shall be continued to 90°C and the distillate collected at that temperature measured at laboratory temperature.

From the volume of distillate to 85°C (or 90°C) the percentage by volume of toluene in the sample shall be read on Graph 2 (opposite), that of the benzene being obtained by difference (see Note).

Temperature Corrections:

The specified temperature shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69.

Note:

As different thermometers may give slightly different results because of difference in thermometric lag, it is advisable to check the results of the test by examining mixtures prepared from the pure hydrocarbons.

SERIAL No.: L.B. 23b-38

Benzene, Toluene and Xylene in Certain Mixtures

(After H. G. Colman and E. W. Yeoman, *J. Soc. chem. Ind.*, 1919, 38, 57T.)

Applicability:

The following method is strictly applicable only to mixtures containing benzene, toluene and xylene within the limits of 50 to 75 per cent of toluene and about 14 to 33 per cent of benzene, the remainder being xylene.

Mixtures of these substances outside the above range may be examined after the addition of an appropriate known amount of pure benzene, pure toluene and/or pure xylene as required.

The test may moreover also be applied to mixtures of these compounds containing paraffins, in which case it shall be assumed that the paraffins are distributed proportionately between the benzene, toluene and xylene.

Apparatus:

A distillation apparatus as described in Serial No. L.B. 6, page 148, except that a standard distillation flask (Schedule No. F2, page 365) shall replace that there specified (Schedule No. F4); the flask shall be fitted with a standard thermometer having the range 70° to 130°C (Schedule No. T8a, page 333).

A standard 100 ml Crow receiver (Schedule No. VI, page 341).

Method:

One hundred millilitres of the sample, measured at laboratory temperature, shall be distilled at the rate of 7 ml per minute (about three drops per second). When the temperature reaches 105°C (see under "Temperature Corrections" below) the flame shall be extinguished, the condenser allowed to drain completely and the volume of the distillate measured at laboratory temperature. The distillation shall be continued at the same rate to 116°C, the flame extinguished, the condenser allowed to drain completely and the volume distilling from 105° to 116°C measured at laboratory temperature. The residue in the flask shall be measured at laboratory temperature and the sum of the three fractions thus obtained shall be at least 99.5 ml.

From the volumes of the first and residual fractions, the percentages by volume of toluene and benzene shall be read from Graph 3 (facing page 186), that of the xylene being obtained by difference (see Note).

If paraffins are present in the sample, their percentage by volume shall be determined according to the method described in Serial No. L.B. 13, page 162; the figure thus obtained shall be deducted proportionately from the percentages of benzene, toluene and xylene previously obtained.

Temperature Corrections:

The specified temperatures shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69.

Note:

As different thermometers may give slightly different results because of difference in thermometric lag, it is advisable to check the results of the test by examining mixtures prepared from the pure hydrocarbons.

SERIAL No.: L.B. 24-38

Xylene in Certain Mixtures

(After D. Northall-Laurie, *Analyst*, 1915, **40**, 384 and P. E. Spielmann and F. Butler-Jones, *J. Soc. chem Ind.*, 1917, **36**, 489.)

Applicability:

The following method was devised for the examination of mixtures of xylene with the hydrocarbons which boil between 139° and 170°C, obtained by fractionating washed benzole or washed solvent naphtha between 138° and 170°C through a 12-bulb pear column. The test is based on the assumption that this fraction behaves as if only two components are present, viz. xylene having a boiling-point of 139°C and a mixture of hydrocarbons boiling at 156°C.

The results must be interpreted with caution.

This test is useful in the examination of solvent naphtha.

Apparatus:

A standard boiling-point flask as specified in Schedule No. B1, page 367, fitted with a reflux condenser in the auxiliary neck of the flask.

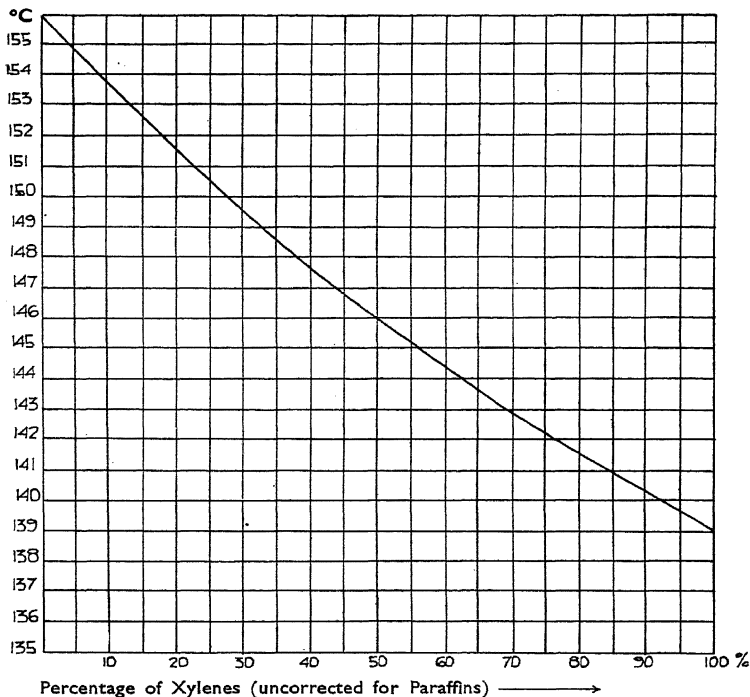
A standard thermometer having the range 130° to 160°C (Schedule No. T12a, page 333) so fitted in the flask that the bulb is entirely within the glass cylinder fused in the main neck of the flask, the bottom of the capillary being level with the lower edge of the side tube joint and the immersion mark being level with the top of the cork.

The flask shall be supported 83 mm above the top of a Bunsen burner, which must be screened from draughts by means of a cylinder of wire gauze. The flask shall rest on a square of asbestos having a central hole 32 mm in diameter. The thermometer shall be screened from convection currents by means of a square of cardboard slipped over the cork in the neck of the flask.

The apparatus shall be well protected from draughts.

Method:

Fifty millilitres of the sample shall be measured into the flask and the apparatus assembled as described. The rate of heating shall be such that the condensate runs back at the uniform rate of one drop per second. The most convenient



Graph 4.—Xylene in Certain Mixtures (Serial No. L.B. 24).

method for regulating this rate is by using a metronome. When the temperature has become constant—which occurs after about ten minutes—it shall be read and corrected for barometric pressure according to the instructions on pages 64 to 69 (see Note ii). Reference of the corrected boiling-point to Graph 4, above, gives the percentage of xylenes (uncorrected for paraffins) in the sample.

The percentage of paraffins shall be determined according to the method described in Serial No. L.B. 13, page 162, on the distillate from 138° to 142°C in the fractionation of a portion of the sample through a standard 12-bulb column (Schedule No. A2, page 370); the figure obtained shall be deducted from that read from Graph 4, to give the percentage of xylenes.

Notes:

(i) If a portion of the sample should distil below 138°C in the fractionation test, the xylenes in that portion may be determined by the method described in Serial No. L.B. 23*b*, page 184.

(ii) In the determination of the boiling-point, the specified conditions must be followed precisely, as the method is particularly sensitive to conditions of working.

SERIAL No.: L.B. 25-38

Benzene, Toluene, and Xylene in Commercial Toluole

(G. Claxton and G. B. Jones.)

Apparatus:

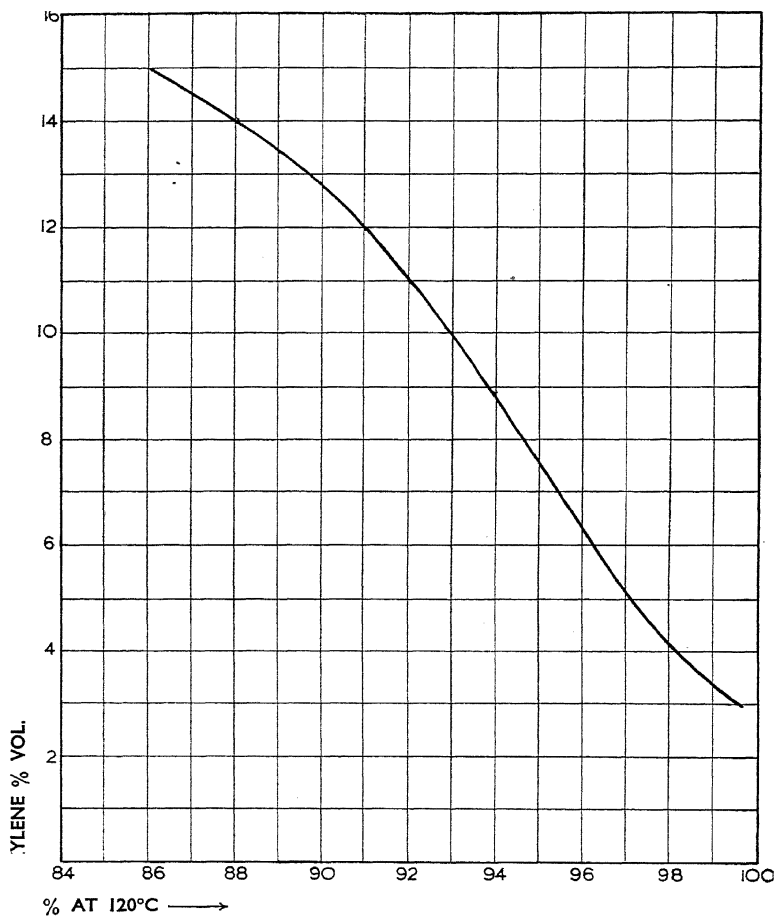
A standard distillation apparatus as described in Serial No. L.B. 6, page 148; the flask shall be fitted with a standard thermometer having the range 70° to 130° C (Schedule No. T8*a*, page 333).

Method:

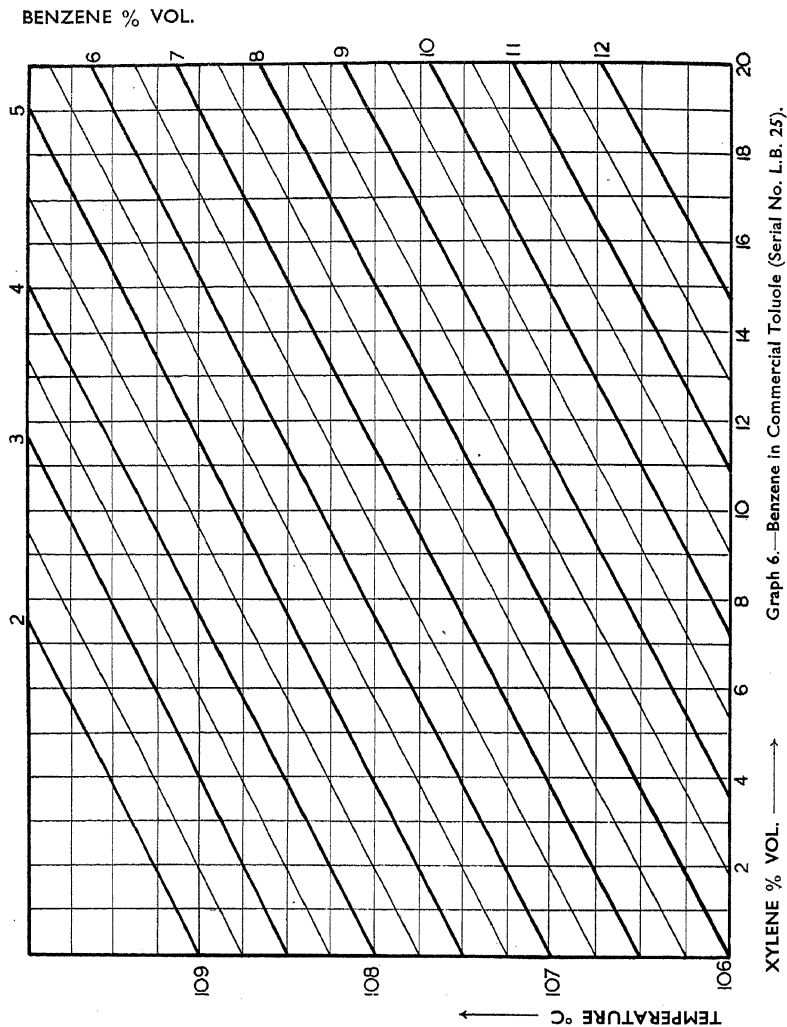
One hundred millilitres of the sample shall be distilled according to the method described in Serial No. L.B. 6, page 148. The temperature at which 5 ml have distilled shall be read as a running-point and the volume of distillate at 120°C (stop-point) shall also be ascertained (see under "Temperature Corrections" below).

The percentage of xylene shall be ascertained from Graph 5, page 189. This percentage shall be referred to Graph 6, page 190; the intersection of the line representing the percentage of xylene and the line representing the temperature at which 5 *per cent* of distillate is collected indicates on the parallel inclined lines the percentage of benzene in the sample. The percentage of toluene shall be obtained by difference (see Note).

If paraffins be present in the sample, their percentage by volume shall be determined according to the method described



Graph 5.—Xylene in Commercial Toluole (Serial No. L.B. 25).



in Serial No. L.B. 13, page 162, and the figure thus obtained shall be deducted proportionately from the percentages of benzene, toluene, and xylene previously obtained.

Temperature Corrections:

The recorded and specified temperatures shall be corrected for barometric pressure according to the instructions on pages 64 to 69.

Note:

As different thermometers may give slightly different results because of difference in thermometric lag, it is advisable to check the results of the test by examining mixtures prepared from the pure hydrocarbons.

SERIAL No.: L.B. 26-38

Metaxylene in Xylole

(After H. P. Reichel, *Chemiker-ztg.*, 1931, 55, 744.)

General:

The material is treated with nitric acid under controlled conditions; the resulting trinitrometaxylene is crystallized from an acetone solution and weighed, allowance being made for the material remaining in solution.

Method:

Exactly 5 ml of the sample shall be measured into a 500 ml conical flask and 20 ml of glacial acetic acid shall be added, followed by 25 ml of 94/95 *per cent w/w* nitric acid (sp.gr. about 1.5) added slowly in the cold. The flask shall be shaken and cooled from time to time in ice water while 40 ml of 98 *per cent w/w* sulphuric acid are added. The mixture shall then be allowed to stand for half an hour at room temperature, after which it shall be heated for 1½ to 2 hours on a boiling water-bath. The flask and its contents shall be cooled in ice water and 450 ml of cold water added. After standing a further half hour in ice water, the crystalline mass which separates shall be filtered through a Jena glass crucible No. 2 G3, and washed with small quantities of water to remove the acid, using 150 ml of water in all.

The crystalline solid shall be sucked as dry as possible on the filter and transferred to a weighed 200 ml conical flask. A reflux condenser shall be attached and the material digested

with 100 ml of acetone for half an hour on a boiling water-bath. The resulting solution shall be cooled for half an hour in ice and shaken occasionally in order to induce crystallization. The liquid shall be brought to 15°C, the trinitrometaxylene filtered through a weighed Jena glass filtering crucible No. 2 G3 and the material in the crucible sucked as dry as possible. The crucible and contents shall be dried for one hour in an oven at 95°C and the weight of the trinitrometaxylene obtained by difference. The flask, which may contain a little adherent crystalline material, shall also be dried and weighed and the net weight added to the weight of the main bulk of trinitrometaxylene.

The correction for the solubility of trinitrometaxylene in acetone is 0.6 g under the above conditions. If W be the weight in grams of trinitrometaxylene obtained, the percentage by weight of metaxylene in the sample is $(W + 0.6) \times 100/9.8$.

SERIAL No.: L.B. 27-38

Colman and Yeoman Test

In preparing the earlier tests in this section, the "Colman and Yeoman" test has been freely drawn upon. At the same time it is recognized that the Colman and Yeoman test in its usual form will continue to be required in certain circumstances for the examination of refined lower boiling products; in addition it is prescribed as one basis on which the complete analysis of crude benzole and of light and middle oils might be performed (Serial Nos C.B. 6a, page 123 and L.M. 5d, page 139). The test is therefore reproduced below, exactly as in the 1929 edition of "Standard Methods" except for minor editorial amendments.

BENZENE, TOLUENE, XYLENE, CARBON DISULPHIDE AND PARAFFINS, INCLUDING NAPHTHENES

(After H. G. Colman and E. W. Yeoman, *J. Soc. chem. Ind.*, 1919, **38**, 57T.)

Applicability:

The following method of analysis is only applicable to samples which have previously been washed with sulphuric acid to remove unsaturated hydrocarbons.

Unwashed samples must first be treated with 5 per cent of their volume of 95 per cent *w/w* sulphuric acid according to the

method described in Serial No. C.B. 5, page 121. The loss on washing is noted and allowed for in the calculation of the results.

Method:

PRELIMINARY DISTILLATION

Five hundred millilitres of the washed sample (or not less than 250 ml if 500 ml are not available) shall be measured at laboratory temperature and distilled at the rate of 5 ml per minute (about two drops per second) in a round bottomed flask of suitable capacity and fitted with a standard 12-bulb pear column (Schedule No. A2, page 370) set vertically. The column shall not be lagged in any way. The following fractions shall be collected without interrupting the distillation and measured separately:—

- (a) Up to 90°C
- (b) 90° to 140°C
- (c) 140° to 160°C
- (d) 160° to 190°C
- (e) Residue at 190°C

If the amount distilling below 90°C be less than 45 *per cent* of the total, small amounts of xylene may come over below that temperature. In such cases the fraction first taken shall be "up to 95°C" and the second "95° to 140°C"; the fraction "up to 95°C" shall then be redistilled in the same manner, the fraction "up to 90°C" collected and the residue added to the "95° to 140°C" fraction, this mixture then forming the "90° to 140°C" fraction.

The examination of the various fractions so obtained is carried out as described under Part I below, this procedure being for samples containing little or no carbon disulphide or paraffins; if these substances be present in material amount, corrections must be made according to the methods given under Part II.

I. CARBON DISULPHIDE AND PARAFFINS ABSENT, OR PRESENT ONLY IN VERY SMALL QUANTITIES

Apparatus:

A standard distillation flask (Schedule No. F2, page 365), fitted with a standard thermometer having the range 70° to 130°C (Schedule No. T8a, page 333) and connected to a Liebig condenser which does not hold up liquid.

A receiver consisting of a standard graduated cylinder of suitable capacity.

Method:

(a) Fraction up to 90°C: (This procedure is also available for the direct analysis of 90's benzole; but see Note i).

One hundred millilitres of the fraction, measured at laboratory temperature, shall be distilled at the rate of 7 ml per minute (about three drops per second). When the temperature reaches 85°C (see under "Temperature Corrections" below), the flame shall be extinguished, the condenser allowed to drain completely and the volume of the distillate measured at laboratory temperature. If the sample should contain more than 20 *per cent* of toluene however, the distillation shall be continued to 90°C and that distillate measured at laboratory temperature.

From the volume of distillate to 85°C (or 90°C), the percentage by volume of toluene shall be read on Graph 2, facing page 184, that of the benzene being obtained by difference. From the amounts of benzene and toluene in 100 ml of the fraction, the quantities present in the whole of the fraction are calculated by proportion (see Note ii).

Temperature Corrections:

The specified temperatures shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69. *This applies also to all other distillations subsequently described in this test.*

Notes:

(i) Small amounts of xylene are usually present in commercial 90's benzole and the toluene figure found by the above procedure is then in fact the percentage of toluene *plus* xylene. These small quantities of xylene shall be determined if desired by fractionation of the sample through a standard 12-bulb pear column (Schedule No. A2, page 370) into the two fractions "up to 90°C" and "above 90°C," the first fraction being analysed as above and the second in the manner described under (b) below for the "90° to 140°C" fraction.

(ii) As different thermometers may give slightly different results because of difference in thermometric lag, it is advisable to check the results by examining mixtures prepared from the

pure hydrocarbons. *This applies also to all other distillations subsequently described in this test.*

(b) Fraction from 90° to 140°C: (This procedure is also available for the direct analysis of commercial toluole).

One hundred millilitres of the fraction, measured at laboratory temperature, shall be distilled at the rate of 7 ml per minute (about three drops per second). When the temperature reaches 105°C, the flame shall be extinguished, the condenser allowed to drain completely and the volume of the distillate measured at laboratory temperature.

The distillation shall be continued at the same rate to 116°C, the flame extinguished, the condenser allowed to drain completely and the volume distilling from 105° to 116°C measured at laboratory temperature. The residue in the flask shall be measured at laboratory temperature and the sum of the three fractions thus obtained shall be at least 99.5 ml. The volumes of the first 100 ml taken and of the fractions obtained must be read at the same temperature.

The results shall be referred to Graph 3, facing page 186. This graph only holds for such mixtures of benzene, toluene and xylene as contain not more than 75 per cent of toluene and give more than 5 per cent of distillate below 105°C and more than 5 per cent above 116°C. When the mixture gives figures which shew no reading on Graph 3, the procedure must be modified by taking for analysis a smaller amount than 100 ml and making the volume up to 100 ml by adding known amounts of pure benzene, pure toluene, or pure xylene (boiling from 138° to 143°C). The quantities of these which it is necessary to add will depend upon the composition of the sample and must be chosen according to judgment, but the following may be taken for guidance:—

In the case of the fraction 90° to 140°C obtained in the manner specified above, it is usually sufficient to take 80 ml of the fraction and add 20 ml of toluene. The composition of the original fraction is calculated in the manner shewn in the following example:—

<i>Mixture of 80 ml fraction and 20 ml toluene yielded</i>	<i>Deduct amount added to mixture</i>	<i>Amount in 80 ml of fraction</i>	<i>Amount in 100 ml of fraction 28.0×100 80</i>	<i>Percentage in fraction</i>
Benzene 28.0 ml	0 ml	28.0 ml	$\frac{28.0 \times 100}{80}$	= 35.0
Toluene 56.0 ml	20.0 ml	36.0 ml	$\frac{36.0 \times 100}{80}$	= 45.0
Xylene 16.0 ml	0 ml	16.0 ml	$\frac{16.0 \times 100}{80}$	= 20.0

The volumes of benzene, toluene, and xylene in the total fraction "90° to 140°C" are calculated from the percentage composition of the 100 ml of the fraction. The volume of benzene and that of toluene in the amount of sample taken for analysis is the sum of the volumes of each constituent in the fractions "up to 90°C" and "90° to 140°C."

Notes:

(i) *50/90 Benzole*: In the case of 50/90 benzole, the sample shall be fractionated through a standard 12-bulb pear column (Schedule No A2, page 370) into the portions distilling "up to 90°C" and "above 90°C," each of which shall be analysed separately in the manner described in (a) and (b) above.

(ii) *Toluole*: When commercial toluole gives results which do not fall on the graph, a mixture of 75 ml of the sample with 15 ml of benzene and 10 ml of xylene usually provides a suitable mixture for the distillation. For example, a sample of 75 ml of a commercial toluole to which 15 ml of benzene and 10 ml of xylene had been added, gave 21.8 *per cent* of distillate up to 105°C and 29.0 *per cent* of residue at 116°C; from Graph 3, the sample therefore contained benzene, toluene, and xylene in the proportion given in the last column of the following table:—

<i>Mixture of 75 ml of sample, 15 ml of benzene and 10 ml of xylene yielded</i>	<i>Deduct amount added to sample</i>	<i>Amount in 75 ml of sample</i>	<i>Amount in 100 ml of sample</i>	<i>Percentage in sample</i>
Benzene 21.0 ml	15.0 ml	6.0 ml	$\frac{6.0 \times 100}{75}$	= 8.0
Toluene 60.0 ml	0 ml	60.0 ml	$\frac{60.0 \times 100}{75}$	= 80.0
Xylene 19.0 ml	10.0 ml	9.0 ml	$\frac{9.0 \times 100}{75}$	= 12.0

(c) *Fraction from 140° to 160°C*: This fraction consists entirely of the constituents of solvent naphtha. To the amount of this fraction shall be added the amount of xylene found in the fraction from 90° to 140°C to give the volume of "solvent naphtha free from toluene" contained in the volume of the sample taken for analysis.

Note:

Benzene and toluene in washed solvent naphtha: For the purpose of these determinations, 100 ml of the solvent naphtha shall be

fractionated through a standard 12-bulb pear column (Schedule No. A2, page 370) and the distillate to 138°C collected in a 100 ml receiver. Twenty millilitres of pure benzene shall be added and the quantity of pure toluene necessary to make up the contents to 100 ml. The mixture shall be distilled in the manner already prescribed for commercial toluole and the percentages of benzene and toluene in the mixture ascertained from the percentages distilling "up to 105°C" and "above 116°C," by reference to Graph 3, facing page 186. The percentage of benzene thus found, less the 20 ml added, and that of toluene, less the amount of pure toluene added, give directly the percentages of benzene and toluene respectively in the original solvent naphtha.

(d) Fraction from 160° to 190°C (Heavy naphtha): This fraction consists entirely of the constituents of heavy naphtha and the volume of the fraction shall be taken as that of heavy naphtha in the volume of sample taken for analysis.

II. CORRECTIONS FOR CARBON DISULPHIDE AND PARAFFINS INCLUDING NAPHTHENES, WHEN THESE ARE PRESENT IN MATERIAL AMOUNT

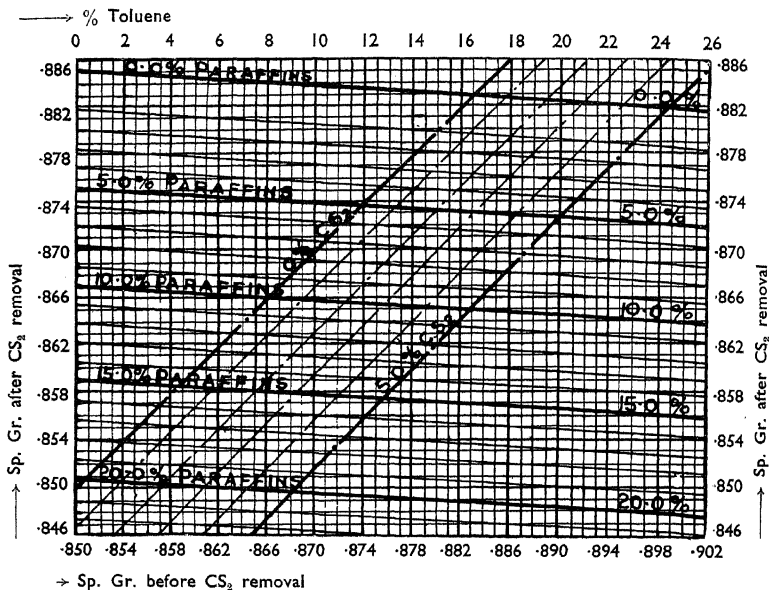
The fraction "up to 90°C" may contain both carbon disulphide and paraffins and the higher fractions may contain paraffins. Corrections in respect of these constituents shall be made by means of specific gravity determinations, as described below:—

(a) Fraction up to 90°C: (This procedure is also available for the direct analysis of 90's benzole.)

The specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the fraction shall be determined by any method giving results accurately to the third decimal place (see Serial No. G.P. 2, page 47). One hundred millilitres of the fraction (or if the quantity available be limited, the combined residue and distillate from the distillation to 85°C or 90°C previously made for determination of the toluene content) shall be mixed with 50 ml of alcohol or industrial methylated spirit free from paraffins and pyridine; 5 g of solid caustic soda shall be added and the whole shaken for five minutes. Two hundred millilitres of water shall be added with renewed shaking; after settlement, the aqueous layer shall be run off, the upper layer washed with two successive quantities of 25 ml of water and dried with calcium chloride. The

specific gravity, $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$, of the dried spirit shall be determined accurately to the third decimal place.

From the specific gravity of the spirit before and after removal of carbon disulphide, the percentage of the latter shall be found from Graph 7, below; from the percentage by volume of toluene previously found in the fraction by distillation to



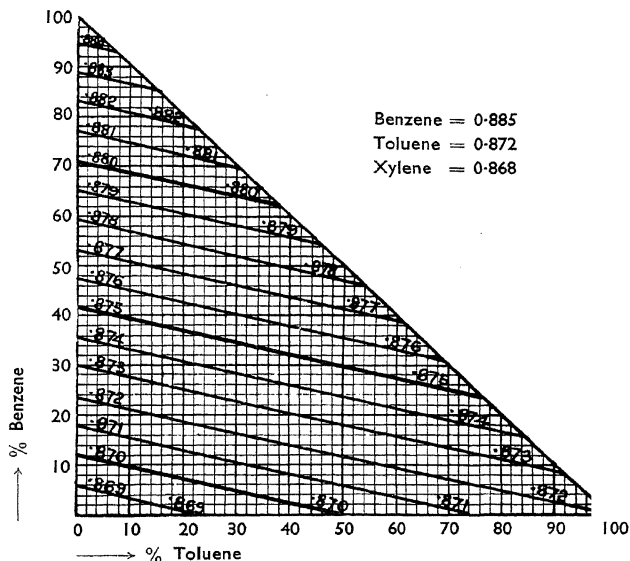
Graph 7.—Carbon Disulphide and Paraffins in Fraction "up to 90°C" and in 90's Benzole (Serial No. L.B. 27).

85°C or 90°C and the specific gravity of the spirit after removal of carbon disulphide, the percentage of paraffins in the fraction is read on the same graph.

(b) Fraction from 90° to 140°C: (This procedure is also available for the direct analysis of commercial toluole.)

The specific gravity, $S_{15.5^\circ\text{C}/15.5^\circ\text{C}}$, of the fraction shall be determined accurately to the third decimal place (see Serial No.

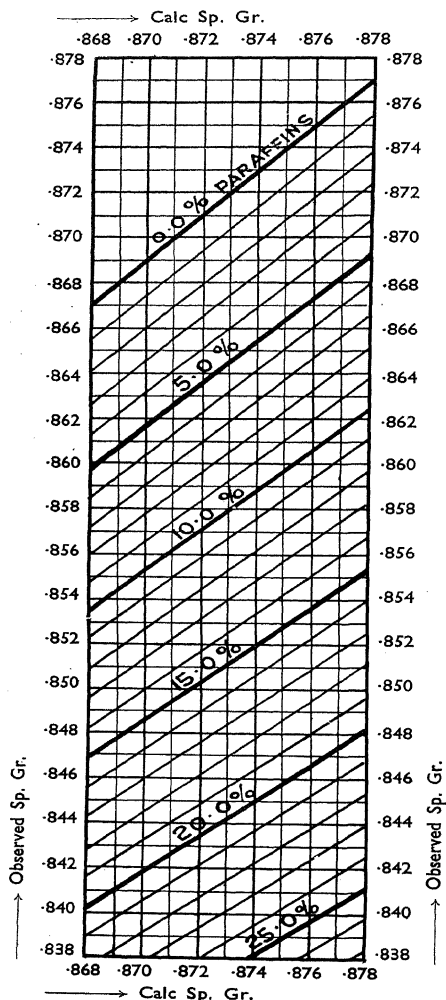
G.P. 2, page 47). Graph 8, below, shall be used to ascertain the calculated specific gravity of a mixture of benzene, toluene, and xylene, free from paraffins and having the percentages of benzene and toluene found in the fraction from the analysis made in Part I above. From the specific gravity of the fraction and the calculated specific gravity, the percentage of paraffins in the fraction shall be read on Graph 9, page 200.



Graph 8.—Calculated Specific Gravity of Paraffin-free Mixtures of Benzene, Toluene and Xylene (Serial No. L.B. 27).

(c) *Fraction from 140° to 160°C.*: Only an approximate estimate of the paraffins in this fraction is possible by specific gravity determinations, because of the unknown composition of the aromatic hydrocarbons. Such an approximation is made on the assumption that the specific gravity of solvent naphtha free from paraffins is 0.868, while that of the paraffins present is

STANDARD METHODS



Graph 9.—Paraffins in Fraction "90° to 140°C"
and in Commercial Toluole (Serial No. L.B. 27).

0.760. The percentage (X) of paraffins shall be found from the equation—

$$0.760 \times X + 0.868 (100 - X) = 100 \times S$$

or—

$$X = \frac{86.8 - 100 \times S}{0.108}$$

where

S = the specific gravity, $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$, of the fraction from 140° to 160°C .

The results thus obtained are usually within 2 to 3 units of the true value and within 1 to 2 units when the paraffin percentage is not more than about 25 (see page 4).

(d) Fraction from 160° to 190°C : Even an approximate estimation of the paraffin percentage by observation of the specific gravity is impracticable, because of the uncertain proportions of coumarone and indene in this fraction. The determination of the paraffin content is best made by the sulphonation method (Serial No. L.B. 13, page 162).



7. CARBOLIC AND CRESYLIC ACIDS

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

Three times the total quantity required for the tests which may have to be applied, shall be supplied.

(a) GENERAL

SERIAL No.: C.C. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: C.C. 2-38

Distillation

Apparatus:

A standard distillation flask (Schedule No. F3, page 365).

A standard thermometer having a suitable range, so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S3, page 373) fitted with a square (150 mm × 150 mm) sheet of wire gauze (about 20 mesh per inch); the flask shall be supported vertically on the gauze.

A standard air condenser (Schedule No. C3, page 370). The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a clamp to hold the condenser.

A standard 100 ml, wide mouthed unstoppered Crow receiver (Schedule No. V1, page 341).

The foregoing is illustrated in Fig. 21, page 204, and represents the arrangement shewn in B.S. 658—1936, Fig. 3, with the optional items particularized.

A Bunsen burner.

Method:

One hundred millilitres of the sample shall be measured at laboratory temperature in the 100 ml receiver and transferred to the distillation flask, the contents of the receiver being allowed to drain for ten minutes into the flask; a fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping; the flask shall be connected to the condenser and the thermometer inserted in the

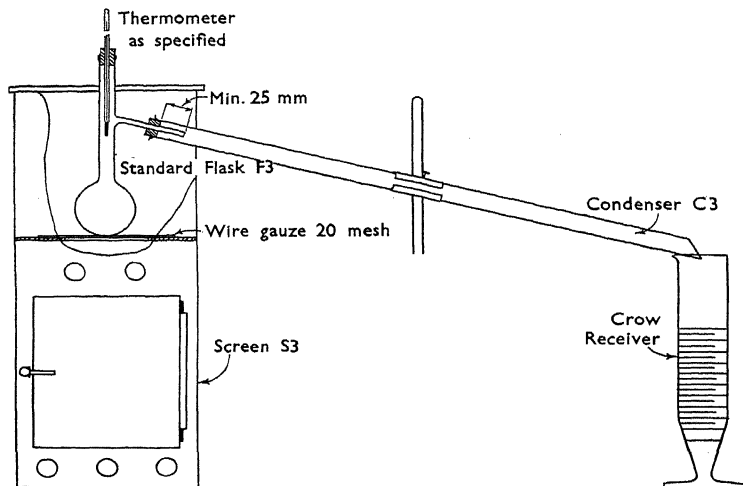


Fig. 21.—Distillation of Carbolic and Cresylic Acids—Assembled Apparatus.

flask. The graduated receiver in which the sample was measured shall be employed, without rinsing or drying, to receive the distillate.

The contents of the flask shall be distilled slowly until all the water has come over (as shewn by the distillate becoming clear at the end of the condenser). The rate of distillation shall then be increased to 3.0 to 3.5 ml per minute (about one drop per second) and unless otherwise required, temperature readings (running-points) shall be taken when 5, 10, 50, 90 and 95 *per cent* by volume of the sample have been collected.

The Bunsen flame shall be extinguished as soon as the temperature reading corresponding to 95 *per cent* of distillate

has been obtained. If other points are required by specification, the corresponding temperatures shall be read without stopping the distillation (running-points), except that if the total distillate be required, the distillation shall be continued until either the dry-point or the pitching-point (see page 63) is reached, when the flame shall be immediately extinguished and the total distillate shall include that which drains from the condenser within five minutes after the flame has been extinguished.

A barometric correction shall be applied to the observed thermometer readings, by allowing 0.05 Centigrade degree for each millimetre by which the pressure differs from 760 mm; the correction is to be added or subtracted, according as the barometric pressure is lower or higher than 760 mm.

SERIAL No.: C.C. 3-38

**Commercial Examination (Water Content, Modified
Lowe's Test, and Residue on Distillation) of
Crude Carbolic Acid**

Applicability:

This test is applicable only to material which gives a crystallizing-point between 45° and 80°F by this method.

Apparatus:

A standard distillation flask (Schedule No. F3, page 365).

A standard thermometer having the range 150° to 250°C (Schedule No. T14a, page 333), so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S3, page 373), fitted with a square (150 mm × 150 mm) sheet of wire gauze (about 20 mesh per inch); the flask shall be supported vertically on the gauze.

An air condenser consisting of a straight glass tube, 750 mm \pm 10 in length and 12.5 mm \pm 0.5 in diameter, with both ends cut at right angles to its axis. The side arm of the flask shall extend 55 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a clamp to hold the condenser.

Three standard Crow receivers (Schedule Nos V1, V2 and V3, page 341) with capacities of 100 ml, 50 ml and 25 ml respectively, the first stoppered. When in use, the receivers shall be vertical and central under the end of the condenser.

The foregoing is illustrated in Fig. 22, below.

A Bunsen burner.

A crystallizing-point apparatus as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having the range 40° to 85°F (Schedule No. T15a, page 333).

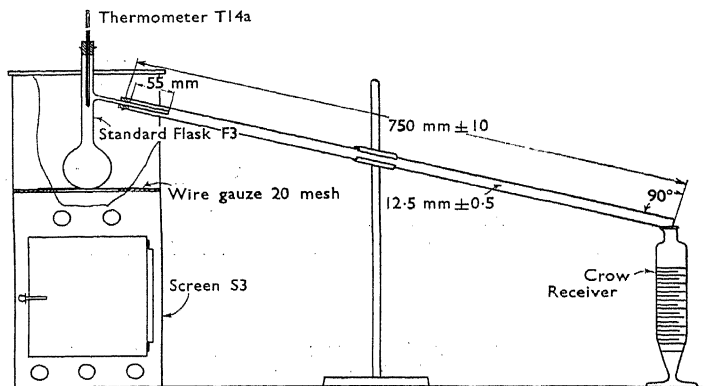


Fig. 22.—Commercial Examination of Crude Carbolic Acid—Assembled Distillation Apparatus.

Method:

All the apparatus shall be *scrupulously* clean and shall be dried thoroughly at the commencement of the determination.

A fragment (about 2 mm cube) of porous or other suitable inert material shall be put in the flask to prevent bumping, and the weight of the flask and the inert material shall be ascertained. One hundred millilitres of the well mixed sample shall be measured at laboratory temperature in the 100 ml receiver and transferred to the flask, the contents of the receiver being allowed to drain for ten minutes into the flask. The distillation apparatus shall be assembled and the distillation carried out in the following manner:—

The first part of the distillation, when water is being distilled, shall be carried out slowly into the 50 ml receiver, at a rate of about one drop in five to six seconds. When all the water has

distilled (as shewn by the distillate becoming clear at the end of the condenser) the distillation shall be continued into the same receiver until 10 ml of clear distillate have collected (see Note i). The 50 ml receiver shall be replaced by the cleaned and dried 100 ml receiver; the rate of distillation shall be increased to one drop per second and continued at this rate until 62.5 ml of distillate have been collected in the 100 ml receiver (see Note i). The 100 ml receiver shall be removed and stoppered, and replaced by the 25 ml receiver. The distillation shall be continued to the pitching-point (see page 63). The amount of residue from 100 ml of the sample shall be obtained by weighing. If the weight of the residue be not more than 2 g, the sample shall be deemed to be free from residue.

At the end of the distillation, the volume of aqueous distillate shall be read in the 50 ml receiver, which must not have been shaken at any stage, and the number of millilitres of aqueous distillate so measured shall be reported as the percentage by volume of water in the sample.

The distillate of 62.5 ml in the 100 ml stoppered receiver (warmed gently if its contents should tend to crystallize) shall be mixed thoroughly by shaking and about 20 ml of the fraction placed in the inner tube of the crystallizing-point apparatus. The assembled apparatus shall be placed in the cooling bath which shall not be more than three Fahrenheit degrees below the expected crystallizing-point, otherwise erroneous readings may be obtained. The contents of the inner tube shall be allowed to cool to about half a Fahrenheit degree above the expected crystallizing-point as ascertained in a previous rough determination on a separate portion of the distillate, and seeded with a small crystal of phenol. The cooling shall be continued with constant stirring until the sample is crystallized throughout.

The highest temperature recorded by the thermometer after crystallization has commenced shall be reported as the crystallizing-point (see Notes ii and iii).

Notes:

(i) The 10 ml and 62.5 ml portions of the distillate shall be measured as they collect and no allowance shall be made in order that the fractions measure 10 ml and 62.5 ml at laboratory temperature. A careful observance of distillation rates will minimize any errors which might arise from this practice.

(ii) If for any reason a second determination of the crystallizing-point be required, whether it be an approximate or an exact determination; it shall be carried out on a fresh portion of the 62.5 ml fraction and on no account on any portion which has previously been used in this part of the examination.

(iii) In the case of normal samples, as the name "crude carboic acid" indicates, the crystallizing-point is assumed to indicate the presence of phenol. As orthocresol alone or possibly naphthalene may give crystallizing-points within the normal range of this test, it is necessary in doubtful cases to confirm the presence of phenol. This may be done by the addition of a small quantity of pure dry phenol which should raise the crystallizing-point when the determination is repeated.

SERIAL No.: C.C. 4-38

Commercial Examination (Water Content, Phenol Content and Residue on Distillation) of Crude Cresylic Acid

Applicability:

This method is strictly applicable only when neutral oils and tar bases are absent, but for practical purposes may be used when the total amount of these substances does not exceed 3 per cent. It is applied when the crystallizing-point as determined by the method described in Serial No. C.C. 3, page 205, is below 45°F.

Apparatus:

A round bottomed flask, having the following dimensions: Bulb capacity: 850 ml \pm 20; length of neck: 100 mm \pm 5; internal diameter of neck: 25 mm \pm 1.

A second round bottomed flask, having the following dimensions: Bulb capacity: 350 ml \pm 10; length of neck: 100 mm \pm 10; internal diameter of neck: 25 mm \pm 1.

A standard 12-bulb pear column (Schedule No. A2, page 370). The column shall not be lagged in any way.

A standard thermometer having the range 150° to 250°C (Schedule No. T14a, page 333) shall be used for the fractionation process and shall be so fitted to the column that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S2, page 373).

A standard air condenser (Schedule No. C3, page 370). The side arm of the column shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a ring and clamp to support the flask, a square (150 mm \times 150 mm) of wire gauze (approximately 20-mesh) being interposed between the ring and the flask.

A second retort stand provided with a clamp to hold the condenser.

A 50 ml stoppered Crow receiver (Schedule No. V2, page 341).

A 250 ml conical flask.

A Bunsen burner.

A standard distillation flask (Schedule No. F3, page 365).

A crystallizing-point apparatus as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having the range 15° to 45°C (Schedule No. T5a, page 333).

Method:

Water

A fragment (about 2 mm cube) of porous or other suitable inert material shall be put into the 850 ml flask to prevent bumping and the weight of the flask and the inert material shall be ascertained. Two hundred and fifty grams of the well-mixed sample shall be weighed into the flask. The flask shall be fitted to the column, to the side arm of which shall be attached the air condenser, and in the neck of which shall be fitted the thermometer, Schedule No. T14a, as indicated under "Apparatus." The assembly of the distillation apparatus shall be completed, the column being set vertically. The contents of the flask shall be distilled slowly into the Crow receiver to 170°C; the phenols in the distillate shall be salted out by the addition of powdered sodium chloride in just sufficient quantity to saturate the aqueous layer and leave a *few* particles undissolved. The aqueous layer shall be measured and the phenols separated; the volume (V ml) of the aqueous layer, multiplied by 0.9 shall be noted as the water content of the 250 g of sample examined, whence (the density of the water being assumed to be 1.00 g/ml) the percentage by weight of water in the sample is 0.36V.

The contents of the flask shall be allowed to cool to about 100°C and the phenols separated from the distillate to 170°C shall be returned to the flask. The distillation shall be

continued at the rate of 3.0 to 3.5 ml per minute (about one drop per second) and fractions taken separately as follows:—

Fraction A	..	up to 195°C, and
Fraction B	..	between 195° and 205°C,

195°C being a running-point and 205°C being a stop-point. Fraction A shall be collected in the weighed conical flask, and fraction B in the 350 ml round bottomed flask.

RESIDUE ON DISTILLATION

The column shall be removed and the flask shall be fitted with a cork carrying a suitable thermometer (e.g. Schedule No. T4a, page 333) and a glass bend connecting the flask directly to the air condenser. The thermometer shall be so fitted that the immersion mark is level with the top of the cork. The exit tube shall extend 10 mm beyond the bottom of the cork in the flask and at least 25 mm beyond the cork in the upper end of the condenser. The distillation shall be resumed and continued to the pitching-point (see page 63). The weight (R g) of residue from the 250 g of sample shall be obtained by difference. If the weight of the residue be not more than 5 g, the sample shall be deemed to be free from residue; otherwise the percentage by weight of residue in the sample is 0.4R.

PHENOL

Fraction B shall be redistilled from the flask in which it was collected, at the same rate and through the same column, and the distillate to 197°C (stop-point) shall be collected in the conical flask already containing fraction A. The combined distillate shall be mixed thoroughly and weighed (W_1) and about 20 g shall be weighed accurately (W_2) into the standard distillation flask (Schedule No. F3). To this shall be added an accurately weighed amount (W_3) of phenol (99/100 *per cent w/w*) about equal to $4W_2$. In order that atmospheric exposure shall be reduced to a minimum, the addition of the phenol shall be made by introducing it directly into the flask.

The flask shall be fitted with a cork in the neck and shall be heated until 2 ml of the distillate have been collected, using no condenser but allowing the side arm of the flask to act in this capacity. The portion of the neck of the flask above the side arm shall be heated gently during the operation to prevent condensation of water in this region, a spirit torch or independent gas burner being used for the purpose. The (cleaned)

50 ml Crow receiver shall be used to receive the distillate. The purpose of this distillation is to remove any final traces of water which may be present, without materially changing the composition of the mixture.

The mixture shall be allowed to cool with a calcium chloride tube fitted to the side arm of the flask and the crystallizing-point shall be determined by the method described in Serial No. C.C. 8, page 217, using about 20 g of the mixture for the test. The fractional phenol content (P) shall be ascertained by reference to Table 25, below.

TABLE 25: RELATION BETWEEN CRYSTALLIZING-POINT AND PHENOL CONTENT

Crystallizing-point °C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
	FRACTIONAL PHENOL CONTENT BY WEIGHT									
27	0.757	0.759	0.760	0.762	0.764	0.766	0.767	0.769	0.771	0.773
28	0.774	0.776	0.778	0.780	0.781	0.783	0.785	0.787	0.788	0.790
29	0.792	0.794	0.795	0.797	0.799	0.801	0.802	0.804	0.806	0.808
30	0.809	0.811	0.813	0.815	0.816	0.818	0.819	0.821	0.823	0.825
31	0.826	0.828	0.830	0.832	0.833	0.835	0.837	0.839	0.840	0.842
32	0.844	0.846	0.847	0.849	0.851	0.853	0.854	0.856	0.858	0.860
33	0.861	0.863	0.865	0.867	0.868	0.870	0.872	0.874	0.875	0.877
34	0.878	0.880	0.882	0.884	0.885	0.887	0.889	0.891	0.892	0.894
35	0.896	0.898	0.899	0.901	0.903	0.905	0.906	0.908	0.910	0.912
36	0.913	0.915	0.917	0.919	0.920	0.922	0.924	0.926	0.927	0.929
37	0.931	0.933	0.934	0.936	0.937	0.939	0.941	0.943	0.944	0.946
38	0.948	0.950	0.951	0.953	0.955	0.957	0.958	0.960	0.962	0.964
39	0.965	0.967	0.969	0.971	0.972	0.974	0.976	0.978	0.979	0.981
40	0.983	0.985	0.986	0.988	0.989	0.991	0.993	0.995	0.996	0.998
41	1.000	—	—	—	—	—	—	—	—	—

The phenol used for addition shall be tested for its fractional content (P_1) of pure phenol by carrying out a similar crystallizing-point determination and referring the resulting figure to the same table.

The weight of phenol added to the test portion is P_1W_3 g; the weight in the mixture ($W_2 + W_3$) is $P(W_2 + W_3)$ g; the weight in W_2 is therefore $(PW_2 + PW_3 - P_1W_3)$ g, and the weight in the combined distillate W_1 and consequently in the 250 g of original sample is $W_1(PW_2 + PW_3 - P_1W_3)/W_2$ g.

The percentage by weight of phenol in the original sample is therefore—

$$\frac{0.4W_1(PW_2 + PW_3 - P_1W_3)}{W_2}$$

where

W_1 = the weight of the combined distillate fraction A and the distillate from fraction B up to 197°C;

W_2 = the weight of the portion of this combined distillate used for the crystallizing-point determination;

W_3 = the weight of the phenol added;

P = the fractional content of pure phenol in the mixture used for the crystallizing-point determination; and

P_1 = the fractional content of pure phenol in the phenol added.

SERIAL No.: C.C. 5-38

Neutral Oils and Pyridine Bases

Apparatus:

A distillation apparatus as shewn in Fig. 23, opposite.

The standard separating funnel receiver shewn in Fig. 23 is described in detail in Schedule No. V4a, page 343 (see Note i); in certain circumstances, the alternative separating funnel receiver, Schedule No. V4, page 343, will be required (see Note ii).

Method:

Fifty millilitres of the sample shall be measured at laboratory temperature into the 500 ml round bottomed flask; 85 ml of 27 per cent *w/w* aqueous caustic soda solution ($S_{15.5^\circ/15.5^\circ}$: 1.300) and 100 ml of water shall be added and the whole agitated thoroughly (see Note iii). A fragment (about 2 mm cube) of porous or other suitable inert material shall be added to prevent bumping. The same measuring cylinder shall be employed, without intermediate cleaning or rinsing, for the successive measurement of the sample, solution and water.

The flask shall be connected as indicated. The graduated portion of the receiver (see Note i) shall be filled with water to prevent lodgment of neutral oils there and the apparatus shall be assembled as indicated in Fig. 23. The distillation shall be carried out as rapidly as is compatible with complete condensation. Care must be taken to ensure that, when the distillate is relatively rich in naphthalene, the naphthalene does not

solidify in the condenser. This can be done by distilling rapidly as indicated and if necessary by stopping the flow of cooling water. If the naphthalene content be high, it may be necessary to empty the water jacket of the condenser. The exit end of the tube of the condenser must be kept above the surface of the

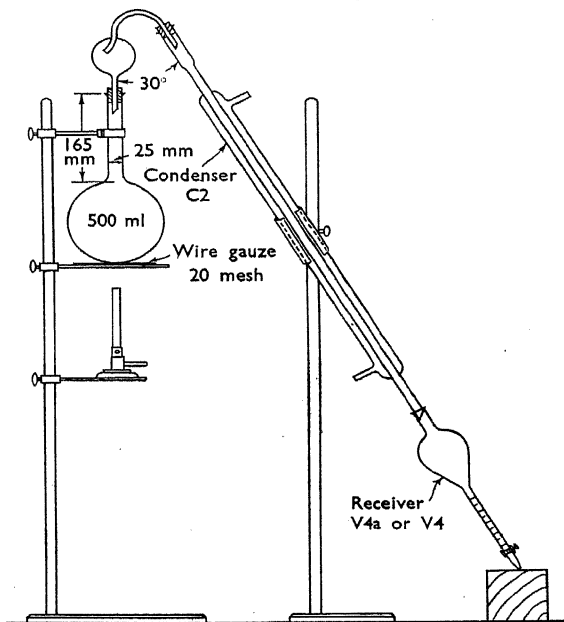


Fig. 23.—Neutral Oils and Pyridine Bases in Carbolic and Cresylic Acids—Assembled Apparatus.

distillate in the receiver, in order to prevent “sucking back” when the distillation is stopped.

All the pyridine bases, naphthalene and neutral oils shall be considered to have distilled when 100 ml of condensate have been collected, but before stopping the distillation the water shall be shut off, the water jacket of the condenser emptied and the condenser tube allowed to steam down for a minute or so.

NEUTRAL OILS

The distillate in the receiver shall be warmed if necessary to a temperature just sufficient to liquefy any solid. The receiver

shall be set vertically and the contents allowed to separate completely, as indicated by clarity of the aqueous layer (see Notes iv and v). The aqueous solution shall be run cautiously into a titration flask which has previously been cleaned from any traces of grease; the volume (V ml) of the neutral oils in the graduated portion of the receiver shall be noted (see Note ii). The percentage by volume of neutral oil in the sample is $2V$ (but see Note iii).

PYRIDINE BASES

The remaining contents of the separating funnel shall be added to the aqueous distillate in the titration flask; the funnel shall be washed with two successive quantities each of 10 ml of distilled water, the washings being added to the titration flask. Three drops of a 0.1 *per cent w/v* solution of phenolphthalein in a 50/50 alcohol/water mixture shall be added. If a purple colour should appear, the mixture shall be titrated drop by drop with 0.1N hydrochloric acid until it is just colourless, and this liquid—or if no purple colour should develop on the addition of the phenolphthalein, the liquid after that addition—shall be titrated as follows:—

Two drops of a 0.1 *per cent w/v* solution of methyl orange in distilled water shall be added to the whole of the distillate (just acid to phenolphthalein) and titration shall be carried out against Normal hydrochloric acid until a pink colour persists, the mixture being well shaken after each addition of acid. The acid shall be added drop by drop, especially at first, and if only one drop of acid be required, pyridine bases shall be reported as nil. If A ml of Normal hydrochloric acid are used, the percentage (*w/v*) of pyridine bases, expressed as pyridine, in the sample is $0.158 A$ (but see Note iii).

Notes:

(i) It is essential that the separating funnel receiver be scrupulously clean and free from grease at the commencement of the test.

(ii) If more than 1 ml of neutral oil be obtained in the receiver Schedule No. V4a, the test shall be repeated on a fresh portion of the sample and the alternative larger receiver, Schedule No. V4, shall be used.

(iii) In the case of samples of high grade products, containing less than 0.1 *per cent* of neutral oils or less than 0.05 *per cent* of pyridine bases, 100 ml of sample with 170 ml of caustic soda

and 100 ml of water shall be used, and the necessary alterations made in the calculation of the results.

(iv) Samples are occasionally met with, the aqueous distillate from which contains oils which will not separate. In such cases, and in such cases only, sodium chloride may be added in successive increments of 5 g until the oil rises.

(v) Naphthalene will be lost by sublimation if the aqueous distillate be allowed to stand for too long a time or at too high a temperature.

SERIAL No.: C.C. 6-38

Acidity and Alkalinity

Applicability:

The following method is intended for the determination of mineral acid (recorded as sulphuric acid) or alkali (recorded as sodium hydroxide) in crude cresylic and crude carbolic acids.

Indicator Solution:

Phenol red indicator solution, prepared by dissolving 0.02 g of solid phenol red in 0.57 ml of 0.1N aqueous caustic soda solution, diluting with a small quantity of alcohol and making up to 100 ml with 20 per cent v/v alcohol.

Method:

One drop of the well shaken sample shall be taken on a glass rod and added to three drops of the phenol red indicator solution on a white porcelain tile. The sample shall be well mixed with the indicator solution and the colour noted. If the colour be yellow (i.e. the sample is acid), the test shall proceed as under part A below; if violet-red (i.e. the sample is alkaline), the test shall proceed as under part B below.

PART A. ACID SAMPLES

One hundred millilitres of the sample shall be measured at laboratory temperature into a 250 ml conical flask. Normal aqueous caustic soda solution shall be run in drop by drop with good shaking and the mixture tested by taking out a drop on a glass rod and mixing it with three drops of the indicator solution on a tile. The addition of caustic soda solution shall be stopped as soon as the indicator turns violet-red. If V_1 be the number of millilitres of Normal caustic soda solution used, the percentage (w/v) of free and combined acid (as sulphuric acid, H_2SO_4) is $0.049 V_1$.

PART B. ALKALINE SAMPLES

One hundred millilitres of the sample shall be measured at laboratory temperature into a 250 ml conical flask. Normal hydrochloric acid solution shall be run in drop by drop with good shaking and the mixture tested by taking out a drop on a glass rod and mixing it with three drops of the indicator solution on a tile. The addition of hydrochloric acid shall be stopped as soon as the indicator turns yellow. If V_2 be the number of millilitres of Normal acid used, the percentage (w/v) of alkali (as sodium hydroxide, NaOH) is $0.04 V_2$.

(b) PHENOLS AND REFINED CRESYLIC ACIDS

SERIAL No.: C.C. 7-38

Water

Apparatus:

A 100 ml graduated receiver.

A glass flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6 or V6a, pages 349 to 356, according as the sample is expected to contain more or less than 1.5 *per cent* of water).

Method:

One hundred millilitres of the sample (see Note) shall be measured at laboratory temperature in the graduated receiver and transferred to the flask; the receiver shall be washed with successive quantities, in all 100 ml, of solvent naphtha (B.S. 479—1938, Part B) which shall be added to the sample in the flask.

The flask shall be attached to the Dean and Stark condensing and collecting system. Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray

tube (see page 351). Finally, the number of millilitres of water in the graduated tube shall be noted at laboratory temperature and reported as the percentage by volume of water in the sample.

Note:

If the percentage of water by weight be required, 100 g of sample shall be used. Assuming the density 1.00 g/ml for the water collected in the graduated tube, the number of millilitres of water collected at laboratory temperature shall be reported as the percentage by weight of water in the sample.

SERIAL No.: C.C. 8-38

Crystallizing-point

Apparatus:

A crystallizing-point apparatus, as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having a suitable range according to the following table:—

<i>Crystallizing-point</i>	<i>Thermometer</i>
Up to 18°C	T1a
Above 18°C up to 42°C	T5a
Above 42°C up to 70°C	T17a

Method:

The inner tube of the apparatus shall be removed from its jacket and a representative sample of about 20 g shall be introduced. A preliminary rapid cooling of the molten mixture shall be carried out to determine the approximate crystallizing-point. The tube shall be partially immersed in a bath about five Centigrade degrees above the crystallizing-point, until all but the last traces of crystal are melted. The inner tube shall be replaced in its jacket and the apparatus assembled as shewn in Fig. 17, page 145, with the cooling bath between six and eight Centigrade degrees below the expected crystallizing-point. Thermometer readings shall be taken at intervals of half a minute with continuous and gentle stirring, this operation being so conducted that seed crystal is present as the temperature of the sample falls to that at which crystallization commences. The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.05 Centigrade degree. If supercooling should take place, the constant temperature may be observed

immediately after the temperature rise. A temperature rise of one Centigrade degree shall be regarded as the maximum allowable.

If a constant temperature be not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second, and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature rise. The point of intersection shall in this case be reported as the crystallizing-point.

SERIAL No.: C.C. 9-38

Residue on Evaporation

Apparatus:

A stoppered U-tube with limbs about 130 mm in length and 12 mm in internal diameter (see Fig. 24, opposite). Each limb is provided with a side tube, either incorporated in the ground glass stopper or (as shewn in Fig. 24) just below the stopper. The stoppers must not be greased.

A copper air bath, having dimensions about 150 mm \times 150 mm \times 150 mm.

A thermometer having an appropriate range (e.g. Schedule No. T4a, page 333).

Method:

The empty U-tube shall be suspended in the bath and heated to $150^{\circ}\text{C} \pm 5^{\circ}$ while a gentle stream of clean air is drawn through the U-tube. The tube shall be allowed to cool with a calcium chloride tube fitted to one side arm, the other being closed; when cool it shall be weighed.

About 10 g of the sample (warmed if necessary to maintain its liquidity) shall be introduced into the bottom of the U-tube and the tube and contents weighed. The apparatus shall be again set up in the air bath with the thermometer also suspended in the bath, the bottom of its bulb being level with the bottom of the U-tube. One of the side limbs shall be connected through the safety flask (see Fig. 24, opposite) to a suction pipe and a gentle stream of clean air shall be drawn through the U-tube.

The air bath shall be maintained at $150^{\circ}\text{C} \pm 5^{\circ}$ until evaporation is complete, when the suction pump shall be disconnected. The tube shall be allowed to cool with a calcium chloride tube fitted to one side arm, the other being closed; when cool it shall again be weighed. The apparatus shall be reassembled and the heating, cooling and weighing procedure repeated until the weight remains constant.

The percentage by weight of residue on the sample shall be calculated and reported.

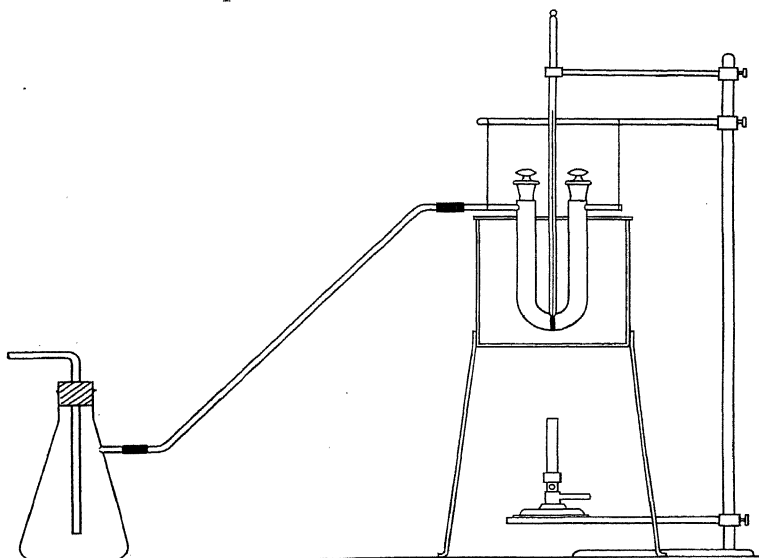


Fig. 24.—Residue on Evaporation of Phenols and Refined Cresylic Acids—Assembled Apparatus.

SERIAL No.: C.C. 10-38

Hydrogen Sulphide

(QUALITATIVE TEST)

Method:

Twenty millilitres of the sample shall be measured into a 100 ml conical flask, over the mouth of which shall be placed a piece of filter paper moistened immediately previously with

a 10 *per cent w/v* aqueous solution of lead acetate. The flask shall be placed on a boiling water-bath and after five minutes the paper shall be examined for coloration.

A coloration deeper than pale yellow indicates the presence of hydrogen sulphide.

SERIAL No.: C.C. 11-38

Solubility of Phenol in Water

Method:

Five grams of the sample shall be weighed in a 150 to 200 ml conical flask of known weight, avoiding the absorption of moisture by undue atmospheric exposure during the weighing. About 50 ml of distilled water, previously boiled and cooled to exactly 15.5°C, shall be added and the flask gently shaken, the temperature being maintained at 15.5°C throughout the test. Further small quantities of the boiled and cooled distilled water shall be added, shaking after each addition, until all oily globules have just disappeared and the solution is clear. The flask and contents shall be weighed; if W g be the weight of the water and phenol, the number of parts of water at 15.5°C required to dissolve one part of the sample is $(W - 5)/5$.

SERIAL No.: C.C. 12-38

Phenol in Liquefied Phenol

Reagents:

0.1N Bromide-bromate solution: The bromide-bromate solution shall be made by dissolving 10 g of potassium bromide and 2.784 g of potassium bromate in distilled water, and making up to 1,000 ml.

Approximately 0.1N sodium thiosulphate solution: The sodium thiosulphate solution shall be made by dissolving 24.82 g of sodium thiosulphate in distilled water and making up to 1,000 ml. This solution shall be standardized immediately before use by means of the 0.1N bromide-bromate solution, as follows:—

Thirty millilitres of the 0.1N bromide-bromate solution shall be run into a 500 ml ground glass stoppered narrow necked bottle and 5 ml of a 20 *per cent w/v* aqueous solution of potassium iodide shall be added, followed by 5 ml of concentrated hydrochloric acid. The stopper shall immediately be placed in position and the contents of the bottle well shaken.

The liberated iodine shall be titrated with the sodium thiosulphate solution until the contents of the flask become only

faintly yellow; starch solution shall then be added and the titration continued until the last traces of blue colour have disappeared.

The factor (n) of the sodium thiosulphate solution is:—

$$\frac{v_1}{v_2}$$

where

v_1 = the number of millilitres of 0.1N bromide-bromate solution used; and

v_2 = the number of millilitres of sodium thiosulphate solution used.

Method:

About 1.5 g of the sample shall be weighed accurately and dissolved in, and made up to 1,000 ml with distilled water. Twenty-five millilitres of this solution shall be run into a 500 ml ground glass stoppered narrow necked bottle, and 30 ml of the 0.1N bromide-bromate solution shall be added, followed by 5 ml of concentrated hydrochloric acid. The stopper shall immediately be placed in position; the bottle shall be shaken repeatedly for half an hour and allowed to stand for a further 15 minutes.

Five millilitres of a 20 *per cent w/v* aqueous solution of potassium iodide shall be added, the bottle shaken thoroughly and the contents titrated by means of the sodium thiosulphate solution, exactly as described in the procedure for standardizing the sodium thiosulphate solution. Any traces of iodine etc. which remain on the stopper during the titration shall be washed into the bottle with distilled water before the titration is completed.

Each millilitre of 0.1N bromide-bromate solution consumed is equivalent to 0.001568 g of phenol; hence the percentage by weight of phenol in the sample is :—

$$\frac{6.272 (V_1 - nV_2)}{W}$$

where

V_1 = the number of millilitres of 0.1N bromide-bromate solution used;

V_2 = the number of millilitres of sodium thiosulphate solution used;

n = the factor for the sodium thiosulphate solution; and

W = the weight of sample taken.

Colour of Cresylic Acid

General:

Cresylic acids may vary in tint from yellow to red shades, and limits are given for each shade of each colour. It should be noted that the colour of cresylic acid darkens on keeping or on exposure to light.

Apparatus:

Two standard 50 ml Nessler cylinders (Schedule No. V10, page 363) similar in every respect including colour.

Standard Solutions:

The several standard solutions shall contain per litre the following quantities of solute/s dissolved in distilled water:—

White	1	0.90 g	Cobalt sulphate
					0.015 „	Potassium dichromate
Very pale	{	Red shade	2R	6.0	„	Cobalt sulphate
				0.015	„	Potassium dichromate
	{	Yellow shade	2Y	4.0	„	Cobalt sulphate
				0.075	„	Potassium dichromate
				1.0	„	Copper sulphate
Pale	{	Red shade	3R	22.5	„	Cobalt sulphate
				0.06	„	Potassium dichromate
	{	Yellow shade	3Y	10.0	„	Cobalt sulphate
				0.18	„	Potassium dichromate
				2.5	„	Copper sulphate
Medium dark	{	Red shade	4R	70.0	„	Cobalt sulphate
				0.5	„	Potassium dichromate
	{	Yellow shade	4Y	320	..	Potassium ferricvanid

The cobalt sulphate used shall be the crystalline heptahydrate; the copper sulphate shall be the crystalline pentahydrate; the potassium dichromate and ferricyanide shall be the anhydrous crystalline compounds. A notification on page 3 requires that all shall be of analytical reagent grade.

The standard solutions keep well, except the last, which shall be freshly prepared on the day of the test; all but the last may be used over a period not exceeding one month from preparation.

Method:

The sample shall be compared with the standard solutions by viewing equal depths in the Nessler cylinders; the colour and shade of the sample shall be reported as not darker than that of the appropriate solution as typified by the description of the depth and shade of colour and/or the index in the table of standard solutions. Any material darker in colour than 4R and 4Y shall be scheduled as Dark—Grade 5.

Note:

The company The Tintometer Ltd has placed on the market a comparator in which depth of colour of the product is compared in succession with coloured glasses mounted in a revolving disc, the glasses matching the standard solutions. This instrument offers a quick and convenient method of determining colour depth.

SERIAL No.: C.C. 14-38

Phenol in Phenol-Cresol Mixtures**Applicability:**

This method is applicable to phenol-cresol mixtures containing 20 per cent or more of phenol (see also Note).

Apparatus:

A crystallizing-point apparatus as shewn in Fig. 17, page 145, fitted with a standard thermometer having the range 15° to 45°C (Schedule No. T5a, page 333).

Method:

Any water shall first be removed from the sample as follows:—

(a) If the amount of water be 1 per cent by weight or less when determined by the method described in Serial No. C.C. 7, page 216, the sample shall be dehydrated by distilling 100 ml in a standard distillation flask (Schedule No. F3, page 365), closing the neck of the flask with a cork and using no condenser but allowing the side arm of the flask to act in this capacity. The portion of the neck of the flask above the side arm shall be heated gently during the operation to prevent condensation of

water in this region, a spirit torch or independent gas burner being used for the purpose.

A standard 25 ml Crow receiver shall be used to receive the distillate and the distillation shall be stopped when 2 ml have been collected. A calcium chloride tube shall be attached to the side arm of the flask and the contents of the flask shall be allowed to cool before withdrawing a portion for the determination of phenol.

(b) If the amount of water be greater than 1 *per cent* by weight when determined by the method described in Serial No. C.C. 7, page 216, about 250 ml of sample shall be placed in an 850 ml round bottomed flask fitted with a standard 12-bulb pear column (Schedule No. A2, page 370) and standard air condenser (Schedule No. C3, page 370). The assembled apparatus shall be adjusted so that the column is vertical. The column shall not be lagged in any way. The contents of the flask shall be distilled slowly until the thermometer indicates 170°C; the phenols in the distillate shall be salted out by the addition of powdered sodium chloride in just sufficient quantity to saturate the aqueous layer and leave a *few* particles undissolved.

The contents of the flask shall be allowed to cool to about 100°C and the phenols separated from the distillate to 170°C shall be returned to the flask. One hundred millilitres of the well mixed contents of the flask shall be treated as described in part (a) above to remove any final traces of water which may be present.

The crystallizing-point of the dried sample resulting from either (a) or (b) above as the case may be shall be determined by the method described in Serial No. C.C. 8, page 217, using about 20 g of the dried sample for the test. If the crystallizing-point be 27.0°C or higher, the fractional phenol content (P) of the dried sample shall be read from Table 25, page 211 (see Note), whence the percentage by weight of phenol in the original sample is—

where
$$P(100 - w)$$

w = the percentage by weight of water in the sample,
as determined by the method described in Serial
No. C.C. 7, page 216; and

P = the fractional phenol content of the dried sample.

If the crystallizing-point of the dried sample be lower than 27.0°C, about 20 g of the dried sample shall be weighed

accurately (W_1) into a standard distillation flask (Schedule No. F3, page 365). To this shall be added an accurately weighed amount (W_2) of phenol (99/100 *per cent w/w*) about equal to $4W_1$. In order that atmospheric exposure shall be reduced to a minimum, the addition of the phenol shall be made by introducing it directly into the contents of the flask.

The flask shall be fitted with a cork in the neck and shall be heated until 2 ml of the distillate have been collected, using no condenser but allowing the side arm of the flask to act in this capacity. The portion of the neck of the flask above the side arm shall be heated gently during the operation to prevent condensation of water in this region, a spirit torch or independent gas burner being used for the purpose. The purpose of this distillation is to remove any final traces of water which may be present, without materially changing the composition of the mixture. The flask shall be allowed to cool with a calcium chloride tube fitted to the side arm and the crystallizing-point of its contents shall be determined by the method described in Serial No. C.C. 8, page 217, using about 20 g of the mixture. The fractional phenol content (P) shall be ascertained by reference to Table 25, page 211 (see Note).

The phenol used for addition shall be tested for its fractional content (P_1) of pure phenol by carrying out a similar crystallizing-point determination and referring the resulting figure to the same table.

The weight of phenol added to the test portion is P_1W_2 g; the weight in the mixture ($W_1 + W_2$) is $P(W_1 + W_2)$ g; the weight in W_1 is therefore $(PW_1 + PW_2 - P_1W_2)$ g, whence the percentage by weight of phenol in the original sample is—

$$\frac{(PW_1 + PW_2 - P_1W_2)(100 - w)}{W_1}$$

where

w = the percentage by weight of water in the sample, determined by the method described in Serial No. C.C. 7, page 216;

W_1 = the weight of the dried sample used for the crystallizing-point determination;

W_2 = the weight of the phenol added;

P = the fractional content of pure phenol in the mixture used for the crystallizing-point determination; and

P_1 = the fractional content of pure phenol in the phenol added.

Note:

The percentage of phenol read from Table 25, page 211, is accurate for ordinary commercial coal tar phenol-cresol mixtures, in which the phenol is present with orthocresol and smaller amounts of meta- and paracresol. If special mixtures, such as phenol with meta- and paracresol are under examination, errors up to ± 1 per cent on the phenol content may arise. In such cases, a special table of crystallizing-points should be prepared, using the same diluent for phenol as is present in the sample under examination (see Dawson and Mountford, *J. chem. Soc.*, 1918, 113, 923).

SERIAL No.: C.C. 15-38

Phenol in Cresylic Acid

(After R. M. Chapin, *United States Department of Agriculture Bulletin*, No. 1308, 22nd November, 1924.)

Applicability:

This method is intended for use when the amount of sample is small; or when the phenol content of the sample is less than 20 per cent; or when the composition of the sample is such that other methods for the determination of phenol are not applicable.

General:

The method is based on the fact that formaldehyde bleaches the colour developed by treating phenol with Millon's reagent, whereas the colour developed by higher phenols with the reagent remains unchanged.

Apparatus:

Two standard 50 ml Nessler cylinders (Schedule No. V10, page 363), similar in every respect including colour.

Four test tubes, about 180 mm in length and about 20 mm in diameter, provided with rubber stoppers. The tubes shall be marked, e.g. by a file scratch, at the 25 ml level.

The Nessler cylinders and test tubes should each carry a further mark so that each may be readily identified; a suggested system for this identification is described under "Method."

A water-bath which may conveniently consist of a beaker, of a suitable size to hold the four test tubes and fitted with a disc of gauze raised slightly from the bottom of the beaker.

Standard volumetric apparatus, including burettes, pipettes and graduated flasks.

Reagents:

Dilute nitric acid: The dilute nitric acid shall be prepared by bubbling air through pure concentrated nitric acid until the acid is colourless and diluting one volume of the acid with four volumes of distilled water. To distinguish it from other strengths of nitric acid used in the test, the solution so prepared is subsequently referred to as "the dilute nitric acid reagent."

Millon's reagent: The Millon's reagent, which is highly poisonous (see Note i), shall be prepared as follows:—

Two millilitres of mercury shall be measured from a burette into a 100 ml conical flask, and 20 ml of pure concentrated nitric acid shall be added. The reaction which results shall be allowed to continue in a fume cupboard, the flask being shaken sufficiently to effect subdivision of the mercury and maintenance of the reaction. When all action has ceased even in the presence of undissolved mercury (about 15 minutes from the start), 35 ml of distilled water shall be added to the contents of the flask. If any separation (of basic mercury nitrate) should occur, the dilute nitric acid reagent shall be added drop by drop until a clear solution is obtained.

A 10 per cent w/w clear aqueous caustic soda solution shall be added drop by drop and with thorough mixing, until the curdy precipitate following the addition of a single drop no longer redissolves, but disperses and produces an apparently permanent turbidity. Five millilitres of the dilute nitric acid reagent shall be added and the contents of the flask shall be thoroughly mixed by shaking.

The Millon's reagent so prepared will remain stable for two days and shall be used for the present test within 36 hours of its preparation; thereafter, the reagent deteriorates and is no longer satisfactory for the purpose of this test.

Standard phenol solution: Stock phenol solution shall be prepared by dissolving a weighed quantity of pure phenol in distilled water to make a solution of a strength not less than 1 per cent w/v. The standard phenol solution shall be prepared on the day of the test, by the addition of sufficient distilled water to a portion of the stock solution to produce a solution containing exactly 0.025 per cent w/v of phenol.

Formaldehyde solution: The formaldehyde solution shall be prepared by diluting 2 ml of the "40 per cent by volume"

formaldehyde solution of commerce with distilled water sufficient to make 100 ml of solution.

Method:

2.5 g of the sample shall be weighed by difference into a 250 ml graduated flask; 10 ml of 10 *per cent w/w* clear aqueous caustic soda solution shall be added and the mixture made up to the 250 ml mark by the addition of distilled water. Five millilitres of this solution shall be measured by means of a pipette into a 200 ml graduated flask; about 50 ml of distilled water and one drop of methyl orange indicator solution shall be added. The dilute nitric acid reagent shall be added drop by drop until neutrality is reached and the contents of the flask shall be made up to the 200 ml mark by the addition of distilled water.

Five millilitres of the resulting solution shall be measured by means of a pipette into each of two of the test tubes, which for the purposes of the present description shall be assumed to have been marked A and B respectively. Into each of the other two test tubes, assumed to be marked C and D, shall similarly be measured 5 ml of the standard phenol solution. Five millilitres of the Millon's reagent (*see Note i*) shall be measured from a burette into each of the four tubes, the contents of each tube thoroughly mixed, and all four tubes placed in the water-bath where they shall remain for *exactly* 30 minutes; the water in the bath shall be maintained at 100°C as nearly as practicable.

At the end of the 30 minutes heating period, the tubes shall be immediately and thoroughly cooled by immersion in a bath of cold water where they shall remain for at least 10 minutes. Five millilitres of the dilute nitric acid reagent shall be added to each tube, and the tubes shaken gently to mix the contents. Three millilitres of the formaldehyde solution, prepared as described under "Reagents" above, shall be added to *one* of each pair of tubes, say to tubes A and C. The contents of all four tubes shall be diluted to the 25 ml mark by the addition of distilled water; the tubes shall be stoppered, well shaken and allowed to stand overnight. The contents of the tubes A and C (to which formaldehyde solution was added) will then be found to possess a yellow colour while the contents of the tubes B and D will possess a red or orange colour.

Twenty millilitres of the contents of the "phenol" tubes C and D shall be measured by means of a pipette into separate 100 ml

graduated flasks, C and D respectively. Five millilitres of the dilute nitric acid reagent shall be added to each flask and the contents of each made up to 100 ml by the addition of distilled water. Two burettes, C and D, shall be filled from the flasks C and D respectively; burette C will then contain the yellow "phenol blank" solution, and burette D will contain the red or orange "phenol standard" solution.

Ten millilitres of the contents of the test tubes A and B, i.e. "sample blank" and "sample unknown" respectively, shall be placed in the Nessler cylinders, A and B respectively. A small quantity of the "phenol standard" shall be run from burette D into the "sample blank" in cylinder A and an equal volume of "phenol blank" shall be run from burette C into the "sample unknown" in cylinder B; the contents of the cylinders shall be agitated thoroughly, rubber stoppers being used if necessary to retain the contents during shaking. The addition of successive equal volumes of "phenol standard" and "phenol blank" from the burettes to the cylinders indicated, with intermediate thorough agitation, shall be continued until the contents of the cylinders are identical in colour (see Note ii). It is essential that the successive additions of "phenol standard" and "phenol blank" to the "sample blank" and "sample unknown" respectively, together with the intermediate agitations and examinations for colour matching, be performed without delay.

Assuming that exactly 2.5 g of the sample were used in the first instance, each millilitre of "phenol standard" used in the colour matching process corresponds to 2 *per cent* by weight of phenol in the sample.

Limits of Accuracy:

The result obtained should fall within ± 3 *per cent* of the actual phenol content of the sample; e.g. if the phenol content of a sample be 10 *per cent*, the figure obtained by the test should lie within 9.7 *per cent* and 10.3 *per cent*.

Notes:

(i) Millon's reagent is highly poisonous and corrosive; in no circumstances must it be drawn into a pipette by means of the unprotected mouth; a burette is consequently specified for its measurement.

(ii) Should the phenol content of the sample be found to exceed 20 *per cent*, the test shall be regarded as a trial only. The colour matching procedure shall be repeated, but instead

of 10 ml of the "sample blank" and "sample unknown" being measured into the Nessler cylinders, equal aliquot portions, made up to 10 ml with a 20/80 mixture of the dilute nitric acid reagent and distilled water shall be used; the aliquot portions shall be chosen so that the amount of "phenol standard" (and "phenol blank") required for matching does not exceed 10 ml.

SERIAL No.: C.C. 16-38

Orthocresol in Cresylic Acid

(After F. M. Potter and H. B. Williams, *J. Soc. chem. Ind.*, 1932, 51, 59t and Standardization of Tar Products Tests Committee, *J. Soc. chem. Ind.*, 1938, 57, 212.)

Applicability:

The following method is applicable to samples containing 40 per cent or more of orthocresol (but see Note i), and is not applicable to certain mixtures which can be prepared but which are not met in commerce.

Apparatus:

A crystallizing-point apparatus, as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having a suitable range according to the following table:—

<i>Crystallizing-point of orthocresol-cineole complex</i>	<i>Thermometer</i>
Below 42°C	T5a
42°C and over	T17a

Method:

Any water shall first be removed from the sample as follows:—

(a) If the amount of water be 1 per cent or less by weight when determined by the method described in Serial No. C.C. 7, page 216, the sample shall be dehydrated by distilling 100 ml in a standard distillation flask (Schedule No. F3, page 365), closing the neck of the flask with a cork and using no condenser but allowing the side arm of the flask to act in this capacity. The portion of the neck of the flask above the side arm shall be heated gently during the operation to prevent condensation of water in this region, a spirit torch or independent gas burner being used for the purpose.

A standard 25 ml Crow receiver shall be used to receive the distillate and the distillation shall be stopped when 2 ml of distillate have been collected. A calcium chloride tube shall be attached to the side arm of the flask and the contents of the flask shall be allowed to cool before withdrawing a portion for the determination of orthocresol.

(b) If the amount of water be greater than 1 *per cent* by weight when determined by the method described in Serial No. C.C. 7, page 216, about 250 ml of the sample shall be placed in an 850 ml round bottomed flask fitted with a standard 12-bulb pear column (Schedule No. A2, page 370) and a standard air condenser (Schedule No. C3, page 370). The assembled apparatus shall be adjusted so that the column is vertical. The column shall not be lagged in any way. The contents of the flask shall be distilled slowly until the thermometer indicates 170°C; the phenols in the distillate shall be salted out by the addition of powdered sodium chloride in just sufficient quantity to saturate the aqueous layer and leave a *few* particles undissolved.

The contents of the flask shall be allowed to cool to about 100°C and the phenols separated from the distillate to 170°C shall be returned to the flask. One hundred millilitres of the well mixed contents of the flask shall be treated as described under (a) above to remove any final traces of water which may be present.

The inner tube of the crystallizing-point apparatus shall be removed from its jacket; 8.40 g of the dry sample and 12.00 g of pure cineole (see Note ii) shall be weighed into it using pipettes drawn off to fine jets.

The contents of the tube, which shall not be exposed longer than is necessary, shall be mixed by means of the glass stirrer until homogeneous. A preliminary rapid cooling of the molten mixture shall be carried out to determine the approximate crystallizing-point. The tube shall be partially immersed in a bath about five Centigrade degrees above the crystallizing-point until all but the last traces of crystal are melted. The inner tube shall be replaced in its jacket and the apparatus assembled as shewn in Fig. 17, page 145, with the cooling bath between six and eight Centigrade degrees below the expected crystallizing-point. Thermometer readings shall be taken at intervals of half a minute with continuous and gentle stirring, this operation being so conducted that seed crystal is present as the temperature of the mixture falls to that at which

crystallization commences. The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.05 Centigrade degree. If supercooling should take place, the constant temperature may be observed immediately after the temperature rise. A temperature rise of one Centigrade degree shall be regarded as the maximum allowable.

If a constant temperature be not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second, and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature rise. The point of intersection shall in this case be taken as the crystallizing-point.

The percentage by weight (C) of orthocresol in the dry sample shall be ascertained (see Note i) either:—

- (a) by reference to Table 26, opposite, which however applies only when the cineole used has a crystallizing-point of 1.2° to 1.4°C; or
- (b) by reference to a table previously prepared by the analyst, using the same cineole as used in the test, together with various mixtures of known orthocresol content.

The percentage by weight of water (w) having been determined by the method given in Serial No. C.C. 7, page 216, and the percentage of orthocresol (C) in the dry sample having been obtained as above, the percentage by weight of orthocresol in the original sample is $C(100 - w)/100$.

Limits of Accuracy:

The result of the test should fall within ± 0.5 unit of the true value (see page 4).

Notes:

- (i) If the first test shews the orthocresol content to be lower than 40 *per cent*, the test shall be repeated, using a mixture of equal weights of the dry sample and of pure orthocresol or of a high grade orthocresol of known orthocresol

content. The purity of the orthocresol employed may be taken from the following:—

<i>Crystallizing-point °C</i>	<i>Purity, per cent</i>
31.0	100
30.7	99.5
30.4	99.0
30.1	98.5

TABLE 26: RELATION BETWEEN CRYSTALLIZING-POINT AND ORTHOCRESOL CONTENT OF ORTHOCRESOL-CINEOLE MIXTURES

Crystal- lizing- point °C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
PERCENTAGE OF ORTHOCRESOL BY WEIGHT										
31	40.0	40.2	40.3	40.5	40.6	40.8	40.9	41.1	41.2	41.4
32	41.6	41.7	41.9	42.0	42.2	42.4	42.5	42.7	42.9	43.0
33	43.2	43.4	43.5	43.7	43.9	44.0	44.2	44.4	44.5	44.7
34	44.9	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.3	46.5
35	46.7	46.9	47.1	47.3	47.5	47.7	47.9	48.0	48.2	48.4
36	48.6	48.8	49.0	49.2	49.4	49.6	49.8	50.0	50.2	50.4
37	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4
38	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.5
39	54.7	54.9	55.1	55.3	55.5	55.7	56.0	56.2	56.4	56.6
40	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6
41	58.8	59.0	59.3	59.5	59.7	60.0	60.2	60.4	60.6	60.8
42	61.0	61.3	61.5	61.7	62.0	62.2	62.4	62.6	62.9	63.1
43	63.3	63.6	63.8	64.0	64.3	64.5	64.8	65.0	65.2	65.5
44	65.8	66.0	66.2	66.4	66.7	66.9	67.2	67.4	67.6	67.8
45	68.0	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.3
46	70.5	70.8	71.0	71.3	71.6	71.8	72.1	72.4	72.6	72.9
47	73.1	73.4	73.7	73.9	74.2	74.5	74.7	75.0	75.2	75.5
48	75.7	76.0	76.3	76.5	76.8	77.0	77.3	77.6	77.8	78.1
49	78.3	78.6	78.9	79.2	79.4	79.7	80.0	80.2	80.5	80.8
50	81.1	81.4	81.7	82.0	82.3	82.6	82.8	83.2	83.4	83.7
51	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7
52	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7
53	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7
54	92.9	93.2	93.5	93.8	94.1	94.4	94.7	95.0	95.3	95.6
55	95.9	96.2	96.5	96.8	97.1	97.4	97.7	98.0	98.2	98.5
56	98.8	99.1	99.4	99.7	100.0	—	—	—	—	—

(ii) It is important to keep the cineole quite dry and away from strong light and the material should be preserved in amber coloured bottles containing a little anhydrous calcium chloride.

SERIAL No.: C.C. 17-38

Metacresol in Cresylic Acid

(After F. Raschig, *Z. angew. Chem.* 1900, **31**, 759.)

Applicability:

The procedure under "Method" below is applicable to cresol mixtures containing (a) not more than 5 per cent of phenol or 10 per cent of xylenols; (b) not less than 45 per cent or more than 80 per cent of metacresol; and (c) not more than 1 per cent of water when determined by the method given in Serial No. C.C. 7, page 216 (see Note i).

Apparatus:

- A 50 ml and a 1,000 ml conical flask.
- A 150 mm porcelain dish.
- A Jena glass filtering crucible, No. 2 G3.

Method:

Exactly 10 g of the sample shall be weighed into the 50 ml flask, 15 ml of concentrated sulphuric acid (containing 96.0 ± 0.2 parts per 100 by weight of H_2SO_4 , checked by titration) shall be added and the contents of the flask mixed. The flask shall be placed in an oven at 100°C where it shall remain for one hour, after which its contents shall be transferred to the 1,000 ml flask. This flask shall be cooled by rotating it for five minutes in a bath of water at 20°C (see Note ii) whereby the liquid sulphonic acid will become deposited as a thick syrup on the walls of the flask.

A quantity of pure nitric acid shall be diluted with distilled water to give a solution of 63.25 ± 1.00 per cent *w/w* strength ($S_{15.5^\circ\text{C}/15.5^\circ\text{C}}: 1.391 \pm 0.005$); 90 ml of this diluted acid at about 20°C (see Note ii) shall be placed in a cylinder. About 20 ml shall be employed to dissolve the sulphonic acid remaining in the 50 ml flask and shall be returned to the measuring cylinder; this operation shall be repeated until the last traces of the

sulphonic acid are removed from the flask. The flask shall then be inverted and drained into the cylinder.

Cautiously, but without delay, the contents of the cylinder shall be transferred to the 1,000 ml flask, the latter being rotated during this operation. Immediately the acid has all been introduced the flask shall be shaken at arm's length until all the sulphonic acid is dissolved. The gentle agitation by swirling shall be continued until brown fumes are copiously evolved. These operations shall be conducted in the fume cupboard and with the mouth of the flask directed away from the operator.

Completion of the nitration process shall be effected by allowing the flask to stand for twenty minutes on an insulating material, such as asbestos, cork or wood, and in a position where it is not exposed to cooling draughts which may tend to chill the mixture (see Note ii). The contents shall be poured into the 150 mm porcelain dish containing 40 ml of distilled water at about 20°C, the flask being thoroughly rinsed, using in all a further 40 ml of distilled water, also at about 20°C. The dish and its contents shall be allowed to stand for two hours at laboratory temperature, when the solid mass shall be reduced to a uniform coarse powder by means of a pestle and transferred as completely as possible to the crucible.

Distilled water, at about 20°C and in a fine stream from a wash bottle, shall be used to transfer the remainder of the powder. The total amount of wash water shall be 100 ml and it must be used in such portions that on transference from the porcelain dish to the crucible each portion will only half fill the crucible. The vacuum shall be gently released before each addition of water to the crucible; as soon as the crucible is half filled the vacuum shall be again connected and the washings allowed to drain before a further quantity of wash water is introduced. When all the solid has been transferred to the crucible, no further washing of the dish is necessary and the contents of the crucible shall be washed by alternately partly filling and then draining, manipulating the vacuum as previously described, until the remainder of the 100 ml of wash water has been used.

The crucible and contents (trinitrometacresol) shall be dried to constant weight (this usually takes from two to three hours) at 95° to 100°C (see Note iii). If W_g be the weight of trinitrometacresol, the percentage by weight of metacresol in the original sample is $10W_g/1.75$.

Notes:

(i) Samples not conforming to any of the requirements under "Applicability" shall before examination by this method be adjusted to conform, in cases under (a) by the addition of a suitable mixture of pure ortho- and pure metacresol; in cases under (b) by the addition of either pure orthocresol to reduce, or pure metacresol to raise, the metacresol content to within the tolerable range; and in cases under (c) as follows:—

About 250 ml of the sample shall be placed in an 850 ml round bottomed flask fitted with a standard 12-bulb pear column (Schedule No. A2, page 370) and a standard air condenser (Schedule No. C3, page 370). The assembled apparatus shall be adjusted so that the column is vertical. The column shall not be lagged in any way. The contents of the flask shall be distilled slowly until the thermometer indicates 170°C; the phenols in the distillate shall be salted out by the addition of powdered sodium chloride in just sufficient quantity to saturate the aqueous layer and leave a *few* particles undissolved.

The contents of the flask shall be allowed to cool to about 100°C and the phenols separated from the distillate to 170°C shall be returned to the flask. The contents of the flask shall be thoroughly mixed and the determination of the metacresol carried out on a portion of the resulting mixture.

The percentage by weight of water (w) having been determined by the method given in Serial No. C.C. 7, page 216, and the percentage of metacresol (C) in the dehydrated sample having been obtained under "Method" above, the percentage by weight of metacresol in the original sample is $C(100-w)/100$.

(ii) Chilling of the reaction mixture or of the nitric acid before use delays the reaction and must be avoided.

(iii) If the trinitrometacresol should tend to melt at or below the temperature of drying, excessive xylenols are indicated. In such circumstances the result shall be discarded and the test repeated after the addition of a known quantity of pure metacresol to the sample.

(c) HIGH BOILING PHENOLS

SERIAL No.: C.C. 18-38

Water**Method:**

The method described in Serial No. C.C. 7, page 216, shall be used.

Phenols**Apparatus:**

A standard 25 ml Crow receiver (Schedule No. V3, page 341).

A standard phenols separating apparatus (Schedule No. V8, page 360).

Method:

Twenty-five millilitres of the sample shall be measured at laboratory temperature in the Crow receiver and transferred to the separating apparatus, allowing the cylinder to drain for ten minutes. Three quantities, each of 25 ml, of clean high boiling neutral petroleum oil (see Note i) shall be measured successively in the same Crow receiver and transferred to the separating apparatus. After draining for ten minutes the level of the mixture shall be adjusted if necessary to the 100 ml mark at laboratory temperature by the addition of more of the high boiling petroleum oil.

The mixture shall be extracted with three successive portions, each of 100 ml, of a 10 *per cent w/w clear* aqueous caustic soda solution. In each case the soda extract shall be run off when the level of the line of separation has remained constant for at least five minutes. Care shall be taken that all visible suspended droplets have cleared from each of the phases before the soda layer is removed. The final volume of the washed oil shall be read at laboratory temperature (see Note ii).

The total contraction, V ml, shall be taken as the volume of the phenols *plus* water, whence the percentage by volume of phenols *plus* water in the sample is $4V$. The percentage by volume of water (w) in the sample shall be determined by the method described in Serial No. C.C. 7, page 216, whence the percentage by volume of phenols in the sample is $4V - w$.

Notes:

(i) Kerosene or high grade diesel oil will be found suitable for employment as the high boiling neutral petroleum oil.

(ii) If appreciable difficulties are encountered because of emulsification, 100 ml of mixture shall be prepared as described under "Method," using a kerosene or diesel oil at least 90 *per cent* of which distills at a temperature higher than the 90 *per cent* distillation-point of the sample of high boiling phenols which is being examined. The mixture shall be distilled to dryness or the pitching-point from a standard distillation flask

(Schedule No. F3, page 365), fitted with a standard air condenser (Schedule No. C3, page 370) and using a naked Bunsen flame. The distillate shall be collected in the standard separating apparatus. It shall be allowed to cool to laboratory temperature and its volume made up to the 100 ml mark with the above special high boiling neutral petroleum oil or with that described in Note (i). The mixture shall be extracted three times with soda as described under "Method" and the percentage of phenols calculated on the assumption that all the phenolic compounds were contained in the distillate.

SERIAL No.: C.C. 20-38

Cresylic Acid—Distillation

(United States Customs Method)

The United States Customs method for the distillation of cresylic acid is frequently used in British laboratories and the Committee has obtained from the Treasury Department of the United States Government permission to include a facsimile reproduction of that method in the present publication (see pages 239 to 245).

FACSIMILE OF U.S. CUSTOMS DISTILLATION
METHOD FOR CRESYLIC ACID

UNITED STATES CUSTOMS

(T. D. 41868)

Cresylic acid—Method of distillation

Following method to be used in distillation of cresylic acid as a substitute for the regulations promulgated in T. D. 41735

TREASURY DEPARTMENT, November 29, 1926.

SIR: The department refers to the regulations promulgated in T. D. 41735 relative to the method used for the distillation of cresylic acid.

Following the publication of the regulations mentioned representations have been made to the department that they do not conform to the methods employed commercially and that neither the Forest Service Hempel flask nor any similar flask with a fractionating column is ever used in testing the coal-tar distillates provided for in paragraphs 27 and 1549 of the tariff act of 1922.

The department is in receipt of a communication from the appraiser at your port, dated the 15th instant, to whom the matter was referred for investigation, in which he states that letters were written by his office to three of the largest domestic manufacturers or distillers of coal tar and three of the largest consumers of both imported and domestic coal-tar acid distillates and that the replies received failed to show that the type of flask with a fractionating column prescribed by T. D. 41735 was used commercially, and the appraiser accordingly expresses the opinion that the Forest Service Hempel flask with beads should be eliminated and that there should be substituted a flask of the type ordinarily used in the trade, modified, however, so as to permit the use of the 3-inch immersion A. S. J. N. thermometer.

After a careful consideration of this question the department concurs in the conclusions reached by the appraiser and accordingly substitutes for the method outlined in T. D. 41735 the following method, which investigation shows conforms to commercial practice:

METHOD FOR THE DISTILLATION OF CRUDE CRESYLIC ACID AND
OTHER COAL-TAR ACID DISTILLATES

APPARATUS

(a) *Flask*.—Made in accordance with Manufacturing Chemists' Association specifications, with dimensions as follows:

Capacity of bulb: 200 c. c.

Total height: 200 mm.

Outside diameter of bulb: 75 mm.

Inside diameter of bulb: 73 mm.

Length of neck: 125 mm.

Inside diameter of neck (max.): 22 mm.

Inside diameter of neck (min.): 20 mm.

Inside diameter of tubulature: 5 mm.

Outside diameter of tubulature: 8 mm.

Length of tubulature: 125 mm.

Length of neck from mouth to center of tubulature: 77 mm.

Angle of tubulature to side neck: 75 degrees.

(b) *Condenser*.—Glass tube air condenser $\frac{1}{2}$ inch $\pm \frac{1}{32}$ inch in diameter and 24 inches long with a flare approximately 4 inches long for making connections with the distilling flask.

(c) *Thermometer*.—A. S. T. N. partial immersion 300° C. thermometer, serial designation D 183-25:

1. These specifications cover a partial immersion thermometer graduated in either centigrade or Fahrenheit degrees as specified, the ranges being -5 to +300° C. or +20 to 580° F., respectively.

2. The thermometer shall conform to the following requirements:

Type: Etched stem, glass.

Liquid: Mercury.

Range and subdivisions: -5 to +300° C. in 1° C. or +20 to +580° F. in 2° F.

Total lengths: 379 to 383 mm. (14.92 to 15.8 in.).

Stem: Plain front, enamel back, suitable thermometer tubing.

Diameter, 6 to 7 mm. (0.236 to 0.275 in.).

Bulb: Corning normal or equally suitable thermometric glass. Length, 10 to 15 mm. (0.39 to 0.58 in.). Diameter, 5 to 6 mm. (0.196 to 0.236 in.).

Distance to 0° C. or 32° F. line from bottom to bulb: 90 to 100 mm. (3.94 to 4.33 in.).

Distance to 300° C. or 572° F. line from top of stem: 25 to 50 mm. (1 to 2 in.).

Filling above mercury: Nitrogen gas.

Top finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: The words "76-mm. immersion" on centigrade thermometers, or "3-in. immersion" on Fahrenheit thermometers and a line around the stem, 75 to 77 mm. (2.96 to 3.04 in.) above the bottom of the bulb, shall be etched on the thermometer.

Special marking: "A. S. T. M.," a serial number and the manufacturer's name or trade-mark shall be etched on the stem.

Scale error: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 1° for the centigrade thermometer or 2° for the Fahrenheit thermometer.

Standardization: The thermometer shall be standardized for 76 mm. (3-in.) immersion and for the following temperatures of the

emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

<i>Thermometer reading</i>	<i>Average temperature of emergent mercury column</i>
50° C. (122° F.)	35° C. (94° F.)
100° C. (212° F.)	49° C. (120° F.)
150° C. (302° F.)	61° C. (142° F.)
200° C. (392° F.)	70° C. (158° F.)
250° C. (482° F.)	76° C. (169° F.)
300° C. (572° F.)	80° C. (176° F.)

Case: The thermometer shall be supplied in a suitable case on which the marking: "A. S. T. M.—5 to 300° C., 76-mm. immersion" or "A. S. T. M. 20 to 580° F., 3-in. immersion," according to the type of thermometer, shall appear.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished thermometer.

The diameter is that measured with a ring gauge.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

(d) *Support for flask*.—Asbestos board with a 1½-inch hole.

(e) *Shield*.—Asbestos board box completely covering the bulb of the flask.

PROCEDURE

Weigh 100 grams ± 0.05 gram (*S*) into the distilling flask and assemble the apparatus, using a cork to connect the tubulature of the distilling flask with the flared end of the air condenser and adjusting the thermometer so that the top of the bulb is on a level with the lower part of the tubulature, the 3-inch or 76-mm. immersion mark on the thermometer is just at the bottom of the regular length No. 10 cork stopper through which the thermometer passes, and the condenser is parallel to the tubulature of the flask. Heat the sample in the flask by means of a burner protected from drafts with a vitrified earthenware guard, and when distillation starts adjust the burner so that approximately 40 drops of distillate are obtained per minute. A cut shall be made at 190° C. and the distillation stopped at 215° C., allowing the contents of the condenser to drain into the tared receiver (a graduated cylinder). These temperatures shall be corrected for the barometric pressure in the laboratory at the time of distillation and any corrections for errors in the thermometer also applied. Directions for barometric corrections follow:

Correction for variation in atmospheric pressure.—Since distillation results are comparable only when obtained under exactly the

same pressure conditions, cresylic acid and other coal-tar distillates shall be distilled at that pressure which, at room temperature, is equivalent to a pressure of 760 mm. of mercury at 0° C. Whenever the atmospheric pressure after correcting to 0° C. is other than 760 mm. a correction must be made. Since alteration of the pressure in the distilling system requires rather complicated apparatus, it is simpler to alter the temperature observation points to correspond to the prevailing pressure.

To determine what the atmospheric pressure at the prevailing room temperature, or at the temperature of the barometer, would be at 0° C., read the barometer and thermometer alongside when about to begin distillation. Calculate the corresponding pressure at 0° C. (P_0) by multiplying the reading of the barometer (P) by the temperature of the barometer (t) and then by the factor 0.0001625, and subtracting the result from the reading of the barometer (P); or, in other words, find the corresponding pressure at 0° C. by means of the following equation:

$$P_0 = P - 0.0001625 Pt.$$

The distilling temperature of cresylic acid is affected plus (+) or minus (-) 0.043° C. for each millimeter variation of the barometer above or below the normal 760 mm. at 0° C. If the barometer reading, after correcting to 0° C. is below 760 mm., the cresylic acid will distill at a slightly lower temperature than under normal pressure. Therefore, the observed temperatures during distillation must be corrected to get their equivalents at normal pressure.

For example, if the barometer reading, after correcting to 0° C. is 750 mm., the correction to be applied at the 190° C. and 215° C. points, respectively, will be 0.043 times 10 mm. = 0.4° C. approximately. The cuts in the distillation will therefore be taken at 189.6° C. and 214.6° C. If the barometer reading corrected to 0° C. was 770 mm. the cuts in the distillation would be taken at 190.4° C. and 215.4° C., respectively.

The distillates obtained below 190° C. and from 190° C. to 215° C., respectively, shall be weighed to ± 0.05 gram (D) and, if the amount obtained in the portion distilling below 190° C. is equal to or more than 5 per cent of the weight of the original sample (S), or that obtained in the portion, or combined portions, distilling below 215° C. is equal to or more than 75 per cent of the weight of original sample (S), analyze it (D) for tar acid content by the liberation method as follows:

LIBERATION METHOD

Transfer approximately 20 c. c. of the portion in either case (or the entire portion in case it amounts to less than 20 c. c.) to a suitable tared Erlenmeyer flask and weigh the portion to ± 0.05 gram (d).

Add approximately 50 c. c. of c. p. xylene and thoroughly mix. Add 25 c. c. of 15 per cent sodium hydroxide solution (or a proportionate amount where the portion taken for analysis is less than 20 c. c.) and heat for 5 minutes at 60–65° C. Transfer the mixture to a separatory funnel of suitable size, shake vigorously, allow the layers to separate clearly and draw off the sodium hydroxide layer. Return the upper layer to the Erlenmeyer flask and repeat the above operation three times using each time the same amount of sodium hydroxide solution as was used the first time. Combine the sodium hydroxide extracts containing the tar acids in a suitable sized beaker and boil with continuous stirring until the solution becomes clear. Five minutes should be sufficient time for the heating.

Cool the solution to room temperature, place the beaker in an ice bath and add slowly, with constant stirring, sulphuric acid, 1–1 (sp. gr. 1.53), until the solution is very slightly acid to litmus. The sulphuric acid must be added slowly to prevent overheating of the solution. The tar acids are thus liberated and form a layer on the aqueous solution.

Transfer the mixture to a special tar acid separatory funnel or a burette of suitable volume and allow to stand until the lower aqueous layer is approximately clear. A portion of this clear aqueous layer may be used for washing all traces of tar acid from the beaker.

Bring the tar acids to a temperature of 20° C. and measure the volume (*t*). Shake the aqueous layer with exactly 20 c. c. of c. p. xylene in a tar acid separatory funnel and read the increase in the volume of the xylene layer, if any (*t'*). This increase in volume is to be considered as tar acid retained by the aqueous layer and the reading of the main tar acid volume increased accordingly.

Determine the specific gravity (*G*) of the liberated tar acids at $\frac{20^{\circ}\text{C.}}{20^{\circ}\text{C.}}$. (A Westphal balance may be used if the quantity of tar acids is sufficient.) Then place approximately 10 c. c. (or as much of the liberated tar acids as is available) in a suitable sized, accurately calibrated glass stoppered cylinder, measure the volume (*V*) of tar acids to tenths of a cubic centimeter, add twice the volume of c. p. xylene, shake thoroughly, allow to stand until the tar acid layer is absolutely clear, and read the volume of separated water, (*W*) if any, to tenths of a cubic centimeter. Calculate the percentage by volume of water thus obtained in the liberated tar acids and deduct the volume of water in the total amount of liberated tar acids corresponding to this percentage from the total volume of tar acids obtained above.

STANDARD METHODS

From the data thus obtained calculate, in terms of the original sample, the percentage of tar acids yielded in the portion analysed by the following formula:

$$\text{Percentage} = \frac{100 DG (t + t^1 - tw)}{d s V}$$

Illustrative numerical example:

S = weight of the original sample = 100 grms.

D = weight of portion distilling below 215°C. = 76 grms.

d = weight of portion taken for the determination of tar acid content = 21 grms.

t = volume of liberated tar acids at 20°C. = 20 c. c.

G = specific gravity of liberated tar acids at $\frac{20^{\circ} \text{C.}}{20^{\circ} \text{C.}}$ = 1.030.

t^1 = volume of tar acids dissolved in the aqueous layer = 0.2 c. c.

V = volume of liberated tar acids taken for the determination of rate content = 10 c. c.

W = volume of water in liberated tar acids taken from the determination of rate content = 0.2 c. c.

Substituting the above values in the formula:

$$\text{Percentage} = \frac{100 \times 76 \times 1.03 \times (20 + 0.2 - \frac{20 \times 0.2}{10})}{21 \times 100} = 73.8.$$

Respectfully,

(110407)

COLLECTOR OF CUSTOMS, New York.

L. C. ANDREWS,

Assistant Secretary.

N.B.—See also amending instructions reproduced on the next page.

(T. D. 42021)

Distillation of cresylic acid

T. D. 41868 relative to the method of distilling cresylic acid amended

TREASURY DEPARTMENT, *March 4, 1927.*

SIR: The department is in receipt of your letter of the 21st ultimo, in which, referring to the thermometers used in the distillation of cresylic acid as outlined in T. D. 41868, you state that you have received a letter from the Bureau of Standards advising you that on repeating your tests with respect to the distillation of cresylic acid as outlined in T. D. 41868 the same results were obtained which corroborated your findings. You accordingly express the opinion that in order to obtain accurate results by the method outlined in T. D. 41868 a correction must be applied to the thermometer readings to compensate for the difference between the stem temperatures prevailing during the distillation and those that occur during the standardization of the thermometer.

In view of your report and in accordance with your recommendation T. D. 41868 is amended by striking out the last two sentences in the first paragraph under "Procedure" reading "These temperatures shall be corrected," etc., and substituting therefor the following:

The observed temperatures shall be corrected for errors in the thermometer and for the differences between the stem temperatures prevailing during distillation and those that occurred during standardization of the thermometer. For room temperatures of 15° C., 25° C., and 35° C. the latter correction is +2.7° C., 2.4° C., and 2.1° C., respectively, and is proportionally larger or smaller for other room temperatures. Corrections shall be applied also for differences in the barometric pressure in the laboratory at the time of distillation as directed below.

Respectfully,
(110407.)

L. C. ANDREWS,
Assistant Secretary.

U. S. APPRAISER OF MERCHANDISE, *New York.*

The facsimile matter on pages 239 to 245, being a photographic reproduction, unavoidably contains the errors in the original American documents; the errors are, however, only editorial.

8. NAPHTHALENE

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least one pound (about 500 g) shall be supplied and shall be stored in an air tight non-absorbent container.

Preliminary Treatment of the Sample:

If the sample consists partly or wholly of lumps, the whole shall be crushed and passed through an 8-mesh sieve (e.g. B.S. test sieve No. 8) and thoroughly mixed before any portion is withdrawn for test.

If the sample should contain free oil, the whole shall be melted in a steam or hot water-bath until completely liquid, care being taken to minimize loss by evaporation. The portion for each test shall be poured from the thoroughly mixed liquid directly into the appropriate apparatus.

(a) CRUDE NAPHTHALENE

SERIAL No.: C.N. 1-38

Water

Apparatus:

A glass flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6 or V6a, pages 349 to 356, according as the sample is expected to contain more or less than 1.5 *per cent* of water).

Method:

One hundred grams of the sample shall be weighed as quickly as possible directly into the flask and 100 ml of solvent naphtha (B.S. 479—1938, Part B) shall be added.

The flask shall be attached to the Dean and Stark condensing and collecting system. Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the

condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray tube (see page 351). Finally, the volume of water in the graduated tube shall be measured at laboratory temperature. Assuming the density 1.00 g/ml, the number of millilitres of water collected shall be reported as the percentage by weight of water in the sample.

SERIAL No.: C.N. 2-38

Crystallizing-point

Apparatus:

A crystallizing-point apparatus as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having the range 65° to 90°C (Schedule No. T7a, page 333).

Material for Test:

While the difference between the crystallizing-point of dry naphthalene and of naphthalene containing a small amount of water is appreciable, the lowering of the crystallizing-point ceases when a minimum of 2 *per cent* of water is present. Therefore, in the absence of instructions to the contrary, if the sample should contain 2 *per cent* or more of water, it shall be examined as received; if the sample should contain a smaller percentage of water, 0.5 ml of water shall be placed in the inner tube of the crystallizing-point apparatus before the sample is introduced as described under "Method" below.

If the crystallizing-point of the dried sample be required, the drying shall be carried out in an atmosphere saturated with the vapours from commercial naphthalene and prepared as described below:—

Anhydrous calcium chloride is placed in the lower compartment of a large desiccator (about 300 mm in diameter and about 270 mm in height) and about 200 g of commercial naphthalene of about the same purity as the sample is spread in a thin layer in flat evaporating dishes placed in the desiccator, the desiccator then being closed for seventy-two hours before the sample to be dried is introduced. The enclosed air thereby becomes saturated with the vapour of naphthalene and with the other volatile substances naturally present in commercial naphthalene.

About 100 g of the sample shall be spread in a thin layer

in a flat evaporating dish and the dish placed in the desiccator prepared as above, where it shall remain for forty-eight hours before withdrawal for test.

If the crystallizing-point of the sample as received be required, there will of course be no preliminary treatment of the sample, whatever may be its water content.

Method:

About 40 g of the material for test (see above) shall be placed in a conical flask (see Note i). The flask shall be loosely stoppered and warmed in a boiling water-bath to 85°C. The contents shall be thoroughly mixed and about 20 g shall be poured into the warmed inner tube of the crystallizing-point apparatus. The tube shall be placed in its jacket and the apparatus assembled as shewn in Fig. 17, page 145, with the bath between six and eight Centigrade degrees below the expected crystallizing-point. The water-bath shall not be heated during the subsequent procedure. Thermometer readings shall be taken at intervals of half a minute, starting when the temperature has fallen to 81°C. The contents of the inner tube shall be stirred continuously and, if water has been added to the sample, stirring shall be sufficiently vigorous to give good mixture of the water and sample.

The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.05 Centigrade degree. Supercooling may occur, in which case the five readings may be observed after the temperature rise. A temperature rise of one Centigrade degree shall be regarded as the maximum allowable.

If a constant temperature within 0.05 Centigrade degree be not obtained over five readings, six final readings shall be taken commencing with the first of two successive readings within 0.05 Centigrade degree. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second, and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the earlier portion of the curve. The point of intersection shall in this case be reported as the crystallizing-point.

If the test has been carried out in the presence of 2 *per cent* or more of water, the addition of the correction 0.85 Centigrade degree to the observed crystallizing-point gives the crystallizing-point of the material on the dry basis. It will be necessary for

the operator to decide whether such a correction has to be made, according to the circumstances (see Note ii).

To avoid ambiguity, the result of the test shall be reported as the—

Uncorrected wet crystallizing-point, if the test has been carried out designedly in the presence of at least 2 *per cent* of water and no correction has been made; or

Corrected wet crystallizing-point, if the correction of 0.85 Centigrade degree has been applied to the uncorrected wet crystallizing-point; or

Dry crystallizing-point, if the test has been carried out on the sample after drying in the prescribed manner; or

Crystallizing-point as received, if the test has been carried out on the sample as received, without regard to the water content of the sample.

Notes:

(i) The method described for filling the sample into the inner tube of the crystallizing-point apparatus caters for samples 20 g of which could not be readily introduced at once. In other circumstances, that quantity may be placed directly into the inner tube if desired.

(ii) If the test be carried out on the sample after drying as described, or if the test be carried out in the presence of 2 *per cent* or more of water and the specified correction be made, the result will be in practical agreement with that obtained by the United States Customs method. That test is carried out on the sample dried as described under "Material for Test" above.

SERIAL NO.: C.N. 3-38

Oil and/or Naphthalene

(W. Kirby)

General:

The approximate percentage of oil, or conversely the approximate percentage of naphthalene in crude naphthalene can be calculated from the crystallizing-point of the sample, or from the depression of the crystallizing-point of pure naphthalene on the addition of a known proportion of the sample.

The crystallizing-points referred to throughout the test are "corrected wet crystallizing-points" (see Serial No. C.N. 2, page 248).

The procedure depends on whether or not the crystallizing-point of the sample is below 70°C.

Method:

The corrected wet crystallizing-point of the sample shall be determined according to the method described in Serial No. C.N. 2, page 248.

PART A. CRYSTALLIZING-POINT AT OR ABOVE 70°C

If the crystallizing-point ($t_1^\circ\text{C}$) so determined be 70°C or higher, the approximate percentage by weight of oil in the sample is $1.96(80 - t_1)$, or alternatively, the approximate percentage by weight of naphthalene in the sample is $1.96t_1 - 56.8$.

PART B. CRYSTALLIZING-POINT BELOW 70°C

If the crystallizing-point of the sample as above determined be below 70°C, about 40 g of pure naphthalene shall be weighed accurately into the conical flask specified in Serial No. C.N. 2; about 10 g of the sample shall be accurately weighed and added to the pure naphthalene in the flask, and the flask loosely stoppered. The flask shall be warmed in a boiling water-bath to 85°C and the contents shall be thoroughly mixed; about 20 g of the mixture shall be poured into the warmed inner tube of the crystallizing-point apparatus.

The apparatus shall be assembled and the corrected wet crystallizing-point ($t_2^\circ\text{C}$) of the mixture determined according to the method described in Serial No. C.N. 2.

The approximate percentage by weight of oil in the sample is—

$$\frac{(W + w)d}{0.51w}$$

or alternatively, the approximate percentage by weight of naphthalene in the sample is—

$$100 - \frac{(W + w)d}{0.51w}$$

where

W = the weight in grams of pure naphthalene used;

w = the weight in grams of sample added; and

d = the depression of the crystallizing-point in Centigrade degrees, and $= 80 - t_2$.

Phenolic Bodies

(After J. J. Fox and A. J. H. Gauge, *J. Soc. chem. Ind.* 1920, 39, 260r.)

General:

The following method is based on the formation of azo dyes when alkaline extracts of phenols from crude naphthalene are mixed with diazotized sulphanilic acid. The colours produced are compared with those produced by standard phenols solutions.

Apparatus:

Two standard 100 ml Nessler cylinders (Schedule No. V9, page 363) similar in every respect including colour.

Standard volumetric apparatus, including pipettes and graduated flasks.

Reagents:

Standard tar phenols solution: The stock tar phenols solution shall be prepared by dissolving one gram of a fraction consisting essentially of xylenols (boiling range 210° to 230°C) in distilled water with 3 ml of 10 per cent w/w clear aqueous caustic soda solution and diluting to 1,000 ml (see Note i). This solution shall be kept stoppered and in a cool dark place.

The standard solution shall be prepared immediately before use by diluting 5 ml of the stock solution to 100 ml with distilled water. This must be discarded at the end of the day.

Sulphanilic acid: 1.91 g of colourless recrystallized sulphanilic acid shall be dissolved in 250 ml of distilled water.

Sodium nitrite solution: 0.85 g of pure sodium nitrite shall be dissolved in 250 ml of distilled water.

Dilute sulphuric acid: One volume of pure concentrated sulphuric acid (sp. gr. 1.84) shall be diluted with three volumes of distilled water.

Caustic soda solution: An 8 per cent w/w clear aqueous caustic soda solution shall be prepared.

Diazotized sulphanilic acid solution: This shall be prepared about five minutes before use, by mixing five volumes of the sulphanilic acid solution with one volume of the dilute sulphuric acid, adding five volumes of the sodium nitrite solution and cooling in ice cold water.

Method:

Fifty grams of the sample shall be dissolved in 150 ml of 90's benzole (B.S. 135—1938, Part D), solution being assisted by heating on the water-bath. The solution shall be transferred to a separating funnel and extracted with two successive quantities, each of 10 ml, of hot 10 *per cent w/w clear* aqueous caustic soda solution. The naphthalene solution shall finally be washed with 20 ml of hot water. The combined washings shall be boiled for ten to fifteen minutes, cooled to laboratory temperature and filtered through ordinary filter paper. The filter paper shall be washed with distilled water and the filtrate, including the washings, shall be diluted to 500 ml in a standard volumetric flask. Five millilitres of this solution shall be diluted to 50 ml when required for the test; this diluted extract is subsequently referred to as the extract A.

Two millilitres of the standard tar phenols solution shall be placed in one of the 100 ml Nessler cylinders with 5 ml of the 8 *per cent w/w* aqueous caustic soda solution and the mixture diluted to 100 ml with distilled water; 10 ml of the diazotized sulphanilic acid solution shall be added and the mixture stirred with a clean glass rod.

Two millilitres of the extract A shall be placed in the second Nessler cylinder and similarly treated with 5 ml of the 8 *per cent w/w* aqueous caustic soda solution, distilled water to make 100 ml, and 10 ml of the diazotized sulphanilic acid. Comparison of the colours produced by the two solutions will determine the amount of the extract A necessary to give a colour identical in depth and shade with that given by 2 ml of the standard tar phenols solution (see Note i). Smaller or larger quantities of the extract A shall then be taken as required to obtain a depth of colour which matches that produced with 2 ml of the standard tar phenols solution. The colour matching shall be completed ten minutes after the addition of the diazotized sulphanilic acid to the Nessler cylinders (see Note ii).

The amount of phenols present in the extract A shall be calculated from the relative quantities of the standard tar phenols solution and of the extract required to produce identical colours on diazotization.

Notes:

(i) In matching the colours, it is important that the shades of colours are the same. The grade of phenols used for the

standard (i.e. xyleneols) covers the boiling-point of naphthalene and should normally give the correct shade of colour (brownish-orange) similar to that produced by the tar phenols extracted from the crude naphthalene. If a yellower orange colour be required, cresylic acid (50/55 *per cent* metacresol) complying with B.S. 521—1938 may be added to the phenols used for the standard, in appropriate quantity to produce a suitable shade. The use of β -naphthol gives a red colour under the conditions of test.

(ii) Several minutes elapse before the maximum shade due to the reaction is reached. On the other hand, if the test solutions are allowed to stand for some hours, the colours frequently alter markedly, probably owing to slow decomposition of the diazonium compound.

SERIAL No.: C.N. 5-38

Matter Insoluble in Toluole

Method:

Ten grams of the sample shall be mixed intimately in a 200 ml beaker with 100 ml of hot 90's toluole (B.S. 805—1938, Part C) (see Note). When all the naphthalene has dissolved, the solution shall be filtered, either (a) through balanced filter papers (e.g. Whatman No. 5) about 150 mm in diameter, or (b) through a weighed Gooch crucible prepared with asbestos and used under vacuum.

If filter papers are used, they must be heated to a temperature between 95° and 110°C and reduced to equal weight by removing the apex of the heavier paper; the uncut paper is used inside the originally heavier paper. The separated material is retained by the inner paper, but both of them are equally subjected to any action exerted by the toluole.

Any insoluble residue shall be transferred to the filter by means of more of the toluole and the filter and contents washed with the solvent until a few drops of the filtrate yield no residue on evaporation. The residue shall be dried in an oven at a temperature between 95° and 110°C until it is of constant weight. The percentage by weight of insoluble matter in the sample shall be calculated and reported.

Note:

The temperature of the toluole throughout the determination must be between 90° and 100°C.

(b) REFINED NAPHTHALENE

SERIAL No.: R.N. 1-38

Crystallizing-point**Apparatus:**

A crystallizing-point apparatus as shewn in Fig. 17, page 145, and described on page 145, fitted with a standard thermometer having the range 65° to 90°C (Schedule No. T7a, page 333).

Method:

About 40 g of the sample, so drawn as to avoid loss of volatile matter, shall be placed in a conical flask (see Note). The flask shall be loosely stoppered and warmed in a boiling water-bath to 85°C. The contents shall be thoroughly mixed and about 20 g shall be poured into the warmed inner tube of the crystallizing-point apparatus. The tube shall be placed in its jacket and the apparatus assembled as shewn in Fig. 17, page 145, with the bath at 75°C. The water-bath shall not be heated during the subsequent procedure. Thermometer readings shall be taken at intervals of half a minute, starting when the temperature has fallen to 81°C. The contents of the inner tube shall be stirred continuously and gently.

The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.05 Centigrade degree. Supercooling may occur, in which case the five readings may be observed after the temperature rise. A temperature rise of one Centigrade degree shall be regarded as the maximum allowable.

If a constant temperature within 0.05 Centigrade degree be not obtained over five readings, six final readings shall be taken commencing with the first of two successive readings within 0.05 Centigrade degree. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second, and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the earlier portion of the curve. The point of intersection shall in this case be reported as the crystallizing-point.

Note:

The method described for filling the sample into the inner tube of the crystallizing-point apparatus caters for samples 20 g of which could not be readily introduced at once. In

other circumstances, that quantity may be placed directly into the inner tube if desired.

SERIAL NO.: R.N. 2-38

Sulphuric Acid Test

Colour Standards:

Colour standards shall be prepared as follows and placed in large test tubes which shall be as nearly identical as possible with each other and with the tube used in the test. Particular care shall be taken that the coloration of the glass is the same in all the tubes and that it is at a minimum.

A stock solution containing 5 g of cobaltous nitrate crystals per 100 ml of solution, and a solution containing 1 g of clean colourless sodium sulphide crystals per 1,000 ml of water, shall be prepared and used as follows:—

<i>Tube No.</i>	<i>Contents</i>
1.	2 ml of the stock cobaltous nitrate solution + distilled water to 10 ml + six drops of the sodium sulphide solution.
2.	6 ml of the stock cobaltous nitrate solution + distilled water to 10 ml + six drops of the sodium sulphide solution.
3.	10 ml of the stock cobaltous nitrate solution + six drops of the sodium sulphide solution.

The six drops of sodium sulphide solution shall be added to each of the tubes from a small pipette.

The standard solutions must be freshly prepared.

Method:

Ten millilitres of water-white, pure, 98/99 *per cent w/w* sulphuric acid, free from nitrogen compounds, shall be heated to 80°C in a large test tube immersed in a water-bath maintained at that temperature. Two grams of the crushed sample of naphthalene shall be added to the contents of the tube, which shall remain immersed in the bath at 80°C and gently shaken for exactly two minutes. The colour thus produced shall be compared immediately with the specified colour standards. The colour obtained with the sample shall be reported as being equal to a particular standard or, if intermediate between two standards, as being darker than one and paler than the next higher standard.

9. TAR BASES

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least one quart (about 1,200 ml) shall be supplied and, as indicated under "Sampling," page 15, the sample shall be stored in a glass stoppered amber coloured or dark blue bottle.

(a) CRUDE TAR BASES

Preliminary Treatment of the Sample:

Any separated aqueous liquor in a sample of crude tar bases shall be removed and the quantity noted, before any portion of the sample is withdrawn for test.

SERIAL No.: T.B. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: T.B. 2-38

Water and Refined Bases

Apparatus:

A round bottomed flask of a size appropriate for the amount of sample available for examination (see first paragraph under "Method").

A standard 12-bulb pear column (Schedule No. A2, page 370). The column shall not be lagged in any way.

A standard thermometer having the range 50° to 210°C (Schedule No. T9a, page 333) so fitted to the column that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S2, page 373).

A standard Liebig condenser with bent end (Schedule No. C1, page 369).

A retort stand provided with a clamp to hold the condenser.

Three standard graduated receivers of appropriate size.

A Bunsen burner.

Method:

At least 250 ml, but preferably 500 ml, of the sample shall be measured at laboratory temperature into the flask (see Note to Serial No. T.B. 5, page 262) and the apparatus assembled with the column vertical. The contents of the flask shall be distilled to 110°C (stop-point) at the rate of 5 ml per minute (about two drops per second).

WATER

The distillate to 110°C shall be cooled to laboratory temperature and its volume noted. It shall be transferred to a stoppered separating funnel of suitable size and 37 *per cent w/w clear* aqueous caustic soda solution (sp. gr. 1.4) shall be added in accordance with the following:—

- (i) If the volume of the distillate to 110°C be 30 *per cent* or less of the volume of the sample taken for the test, twice its volume of the caustic soda solution shall be used; but
- (ii) If the volume of the distillate to 110°C should exceed 30 *per cent* of the volume of the sample taken for the test, three times its volume of the caustic soda solution shall be used.

The contents of the funnel shall be well shaken and allowed to stand until separation is complete. A portion of the caustic soda layer shall be run off and its specific gravity shall be determined. If the specific gravity be below 1.35, the water content is high. The portion of the caustic soda solution withdrawn shall in that case be returned to the separating funnel and sufficient solid caustic soda shall be added to raise the specific gravity of the solution, after further agitation, to at least 1.35. The caustic soda layer shall be completely run off. The volume of the dehydrated distillate shall be noted at laboratory temperature and the loss in volume shall be taken to represent its water content, whence the percentage by volume in the sample shall be calculated.

REFINED BASES

After measurement as above, the dehydrated distillate shall be returned to the residue in the flask and the apparatus reassembled. The contents of the flask shall be distilled at the rate of 5 ml per minute to such temperatures that two

- (a) 90 per cent at 140°C, or 90 per cent at 160°C, depending on the basis on which the results are to be reported; and
- (b) 90 per cent at 180°C.

The following shall be reported, the amount of each constituent being calculated as the percentage by volume on the original sample:—

The two fractions (a) and (b) above shall be examined by the methods laid down for refined pyridine bases (Serial Nos R.P. 1 to 8, pages 263 to 266) and refined heavy bases (Serial Nos H.B. 1 to 3, pages 266 to 267) respectively.

The pyridine bases reported above, while not anhydrous, may contain less water than is normally permitted by commercial specifications and adjustments may be made accordingly.

Neutral Oils

Should the refined pyridine obtained in Serial No. T.B. 2, above, fail to pass the test for miscibility with water (Serial No. R.P. 8, page 266) in accordance with the specification being followed, a determination of the neutral oils, etc. in the crude pyridine may be required, in which case the present test shall be applied.

A distillation apparatus as shewn in Fig. 23, page 213.

The standard separating funnel receiver shewn in Fig. 23 is described in detail in Schedule No. V4a, page 343 (see Note i); in exceptional circumstances, the alternative separating funnel receiver Schedule No. V4, page 343, will be required (see Note ii).

Material for Test:

The operator must decide, according to the end in view, whether the test shall be performed on a portion of the original sample or on a mixture of equal aliquot portions of the two fractions (a) and (b) of refined pyridine and heavy bases obtained in the method described in Serial No. T.B. 2, page 257.

Method:

A quantity of the material for test (see above), at least 50 ml in volume, shall be measured at laboratory temperature into the 500 ml round bottomed flask; a quantity of 25 *per cent w/w* sulphuric acid, equal to three times the volume of the material taken for test, shall be added. One hundred millilitres of water shall be added and the whole agitated thoroughly. A fragment of porous or other suitable inert material may be used to prevent bumping. The same measuring cylinder shall be used, without intermediate cleaning or rinsing, for the successive measurement of the sample, acid and water.

The flask shall be connected as indicated. The graduated portion of the receiver (see Note i) shall be filled with water to prevent lodgment of neutral oils there and the apparatus shall be assembled as indicated in Fig. 23, page 213. The distillation shall be carried out as rapidly as is compatible with complete condensation. If the distillate be relatively rich in naphthalene, care must be taken to ensure that the naphthalene does not solidify in the condenser. The exit end of the tube of the condenser must be kept above the surface of the distillate in the receiver, in order to prevent "sucking back" when the distillation is stopped.

All the neutral oils shall be considered to have distilled when 40 *per cent* by volume of the contents of the flask has distilled, but before stopping the distillation the water shall be shut off, the water jacket of the condenser emptied and the condenser tube allowed to steam down for a minute or so. The distillate in the receiver shall be warmed if necessary

to a temperature just sufficient to liquefy any solid. The receiver shall be set vertically and the contents allowed to separate completely (see Notes iii and iv). The aqueous solution shall be cautiously run off; the volume of the neutral oils in the graduated portion of the receiver shall be noted and the percentage by volume in the sample calculated.

Notes:

(i) It is essential that the separating funnel receiver be scrupulously clean and free from grease at the commencement of the test.

(ii) If more than 1 ml of neutral oil be obtained in the receiver Schedule No. V4a, the test shall be repeated on a fresh portion of the sample and the alternative larger receiver, Schedule No. V4, shall be used.

(iii) Samples are occasionally met with, the aqueous distillate from which contains oils which will not separate. In such cases, and in such cases only, sodium chloride may be added in successive increments of 5 g until the oil rises.

(iv) Naphthalene will be lost by sublimation if the aqueous distillate be allowed to stand for too long a time or at too high a temperature.

SERIAL No.: T.B. 4-38

Ammonia

(T. Callan)

Method:

A suitable amount of the sample, purified if necessary by steam distillation (see Note), shall be weighed accurately, dissolved in distilled water and made up to 250 ml. Fifty millilitres or other suitable aliquot portion of this solution shall be titrated with 0.1N sulphuric acid using 0.5 ml of thymol blue solution (0.04 per cent *w/v* in 60 per cent *v/v* alcohol) as indicator, until first the blue and then the green colour disappears and the final yellow colour matches that of a prepared standard (see below). The amount of ammonia in the sample shall be calculated, 1 ml of 0.1N sulphuric acid being equivalent to 0.0017 g of ammonia (NH_3).

The colour standard shall be prepared by titrating 25 ml of about 0.1N ammonia with 0.1N sulphuric acid, using methyl orange solution as indicator. A separate 25 ml of the ammonia solution shall be neutralized exactly by adding an amount of

the acid equal to that required in the titration; 0.5 ml of the thymol blue indicator solution shall be added to this neutralized ammonia solution, together with pure pyridine, the amount of the latter being about that which is used in the actual determination. No appreciable colour change is usually noted on addition of the pure pyridine. The whole shall be diluted with distilled water to the volume (about 75 ml) used in the titration of the sample and transferred if necessary to a vessel similar to that in which the titration of the sample is performed.

Note:

If it be necessary to carry out the steam distillation, the total steam distillate (i.e. bases *plus* water) shall be made up to 250 ml with distilled water and an aliquot portion titrated as indicated.

SERIAL No.: T.B. 5-38

Ash**Method:**

About 200 g of the sample shall be weighed accurately into a weighed distillation flask and distilled until only a small residue remains, care being taken that coke is not formed (see Note). The flask and contents shall be weighed; about half or, if there be only a small amount of residue in the flask, as much as possible of the well mixed contents shall be transferred to a weighed porcelain or silica crucible and the crucible and contents weighed.

The crucible shall be heated cautiously to expel volatile matter, avoiding ebullition, and finally ignited until all combustible material is burnt. The crucible shall be allowed to cool and shall be weighed. The percentage by weight of ash in the sample shall be calculated and reported.

Note:

Provided there has been no formation of coke in the flask used for the distillation process described in Serial No. T.B. 2, page 257, the residue from that distillation may be used instead of the residue obtained from the distillation described above. In such circumstances it will of course be necessary to weigh the round bottomed flask at the beginning of the method Serial No. T.B. 2, and to note the weight of sample used for that test.

(b) REFINED PYRIDINE BASES**General:**

The sale and purchase of refined pyridine are almost exclusively governed by the German specifications or by slightly varied versions thereof adopted in other countries. While many comments on the 1929 text have been received and would have resulted in improvements, no useful purpose would be served by departing from the requirements of the above mentioned specifications, in the tests in this section.

SERIAL No.: R.P. 1-38

Specific Gravity; Density**Method:**

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: R.P. 2-38

Colour**Apparatus:**

Two standard 50 ml Nessler cylinders (Schedule No. V10, page 363) similar in every respect including colour.

Method:

The colour of the sample shall be compared with that of a solution of 0.1N iodine freshly diluted with distilled water according to the requirements of the specification. Unless otherwise specified, the sample and the diluted iodine solution shall be contained in the Nessler cylinders, 50 ml of the sample and solution being used and comparison being made while the cylinders are held three inches vertically above the surface of an opaque opal glass sheet reflecting diffused daylight.

SERIAL No.: R.P. 3-38

Water**Method:**

Twenty millilitres of the sample shall be shaken with 20 ml of 37 *per cent w/w* clear aqueous caustic soda solution (sp. gr. 1.4) in a stoppered cylinder graduated to 0.1 ml. The contents of the cylinder shall be allowed to stand for six hours. The decrease in volume of the pyridine shall be reported as the

water content of the 20 ml of sample used, or multiplied by 5 to give the percentage by volume of water in the sample, as required.

SERIAL NO.: R.P. 4-38

Distillation

Apparatus:

A flask to the specification represented by Fig. 25, below.

A standard thermometer having the range 50° to 210°C (Schedule No. T9a, page 333) so fitted in the flask that its bulb is in the middle of the expansion in the neck of the flask.

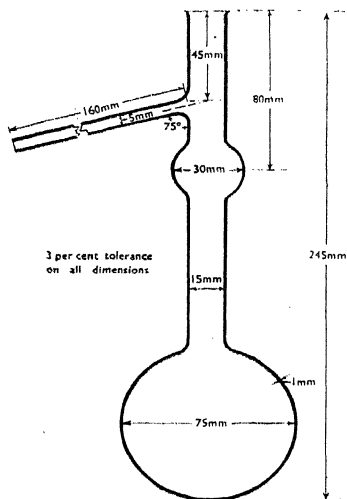


Fig. 25.—Distillation of Refined Pyridine Bases—Distillation Flask.

A standard draught screen (Schedule No. S3, page 373), fitted with an asbestos board having a central hole 30 mm in diameter; the flask shall be placed in position and pressed down to close completely the hole in the asbestos board.

A standard Liebig condenser with bent end (Schedule No. C1, page 369).

A retort stand provided with a clamp to hold the condenser.

A standard 100 ml Crow receiver (Schedule No. V1, page 341).

A Bunsen burner.

Method:

One hundred millilitres of the sample shall be distilled at the uniform rate of 5 ml per minute, measurements of the distillate being taken at the temperatures and under the conditions indicated in the specification which is being followed.

SERIAL No.: R.P. 5-38

Congo Red Test**Stock Solution:**

Exactly 10 ml of the sample shall be measured into a 100 ml graduated flask and the volume made up to the mark with distilled water to form a stock solution which is used for this and the next succeeding tests.

Method:

Normal sulphuric acid solution shall be added from a burette to 10 ml of the stock solution until a drop of the mixture on Congo red test paper produces a distinct blue border which soon disappears. The volume of acid used shall be reported.

The Congo red test paper shall have been freshly prepared by passing filter paper through a 0.1 *per cent w/v* solution of Congo red in distilled water and drying the paper.

SERIAL No.: R.P. 6-38

Cadmium Chloride Test**Method:**

Ten millilitres of the stock solution (see under Serial No. R.P. 5 above) shall be diluted to 100 ml with distilled water. Ten millilitres of the resulting diluted stock solution shall be shaken vigorously with 5 ml of a 5 *per cent w/v* aqueous solution of anhydrous (fused) cadmium chloride. The mixture shall be allowed to stand for ten minutes and shall then be examined for the presence of precipitate, the nature and extent of which shall be noted.

If the specification should require a determination of the weight of this precipitate, the latter shall be filtered, without previous washing, on a dry weighed filter paper, about 90 mm in diameter and 0.45 to 0.55 g in weight. The precipitate shall be dried for one hour at a temperature of 50° to 70°C and weighed. No washing of the precipitate shall be performed at any stage; the filtrate may however be used to transfer the precipitate to the filter paper.

SERIAL No.: R.P. 7-38

Nessler's Reagent Test**Method:**

Five millilitres of Nessler's reagent (see Note) which is not more than one month old and which has been preserved from the direct action of light, shall be added to 10 ml of the diluted stock solution (see under Serial No. R.P. 6 above). The colour of the precipitate shall be noted according to the requirements of the specification.

Note:

Nessler's reagent may be prepared as follows:—

3.25 g of potassium iodide are dissolved in 25 ml of water and 1 ml of the solution is set aside. A cold saturated solution of mercuric chloride is added to the remainder, with constant stirring, until a slight permanent precipitate is formed (about 30 ml are required). The remaining millilitre of potassium iodide solution is added and more of the mercuric chloride solution is cautiously added until a very slight permanent precipitate is formed. A cold solution of 15 g of potassium hydroxide in 30 ml of water is added and the volume made up to 100 ml with water. The mixture is allowed to settle, the liquid is decanted and stored in the dark. The water used at all stages of the preparation must be free from ammonia.

SERIAL No.: R.P. 8-38

Miscibility with Water**Method:**

Twenty millilitres of the sample shall be shaken thoroughly with 40 ml of distilled water at laboratory temperature and the opalescence or otherwise of the solution shall be noted.

(c) REFINED HEAVY BASES

SERIAL No.: H.B. 1-38

Specific Gravity; Density**Method:**

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: H.B. 2-38

Distillation**Method:**

This test shall be performed in the manner prescribed for refined pyridine (see Serial No. R.P. 4, page 264).

SERIAL No.: H.B. 3-38

Solubility in Acid**Method:**

Fifty millilitres of the sample shall be measured into a standard phenols flask (Schedule No. V5, page 347); 120 ml of hydrochloric acid (containing at least 30 *per cent* by weight of HCl) and 30 ml of distilled water shall be added. The contents of the flask shall be well mixed and the solution allowed to stand for four hours. It shall then be examined for homogeneity; the volume of any undissolved portion of the sample may be ascertained by adding saturated aqueous sodium chloride solution to bring the supernatant material into the graduated portion of the flask.

10. CREOSOTE OIL

Applicability:

The following tests are applicable to normal creosote oil, cresylic creosotes, creosote wash oils, horticultural creosotes, creosote fuel oils and the like.

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least a quarter of a gallon (about 1,200 ml) shall be supplied for a complete examination according to the following tests.

Preliminary Treatment of the Sample:

The sample shall be thoroughly mixed immediately before any portion is withdrawn for testing. If it should shew signs of separation of soluble solid constituents, the container shall be immersed in a water-bath to a temperature just sufficient to dissolve them, and portions for test shall be taken from the warmed mixed sample. A naked flame or other source of intense heat shall not be used to warm the sample.

SERIAL No.: C.O. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, page 47, and Note ii on page 55 (specific gravity) or Serial No. G.P. 1, page 31 (density)).

SERIAL No.: C.O. 2-38

Water

Apparatus:

A 100 ml graduated receiver.

A glass flask of about 500 ml capacity, fitted with a standard Dean and Stark condensing and collecting system (Schedule No. V6 or V6a, pages 349 to 356, according as the sample is expected to contain more or less than 1.5 per cent of water).

Method:

A portion of the sample shall be warmed if necessary to the minimum temperature of complete liquidity and after mixing,

100 ml (see Note) shall be measured in the graduated receiver and transferred to the flask; the receiver shall be washed with successive quantities, in all 100 ml, of solvent naphtha (B.S. 479—1938, Part B) which shall be added to the sample in the flask.

The flask shall be attached to the Dean and Stark condensing and collecting system. Heat shall be applied to the flask and so regulated that the condensate falls from the end of the condenser at a rate of two to five drops per second.

The distillation shall be continued until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube and until the volume of water collected remains constant. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation by a few drops per second. Any droplets of water which adhere to the lower end of the condenser tube shall be washed into the receiver with solvent naphtha, using the spray tube (see page 351). Finally, the number of millilitres of water in the graduated tube shall be noted at the temperature at which the 100 ml of sample were measured and shall be reported as the percentage by volume of water in the sample.

Note:

If the percentage of water by weight be required, 100 g of the sample shall be used for the test. Assuming the density 1.00 g/ml for the water collected in the graduated tube, the number of millilitres of water collected at laboratory temperature shall be reported as the percentage by weight of water in the sample.

SERIAL No.: C.O. 3-38

Distillation

General:

The method of distillation described below is in principle that required in British standard practice (B.S. 144—1936) and shall be used in the absence of other instructions (see however, Serial No. C.O. 14, page 294).

Apparatus:

A standard distillation flask (Schedule No. F4, page 365), held in the vertical position by means of a clamp at the extreme upper end of the neck.

A standard thermometer having the range 90° to 400°C (Schedule No. T4a, page 333), so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork (see page 366).

A standard draught screen (Schedule No. S3, page 373), from which the shelf has been removed.

A standard air condenser (Schedule No. C3, page 370). The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

A retort stand provided with a clamp to hold the condenser.

Receivers consisting of weighed 25 ml and/or 50 ml Crow receivers; the smaller size shall be used to receive any distillate which it is known will not exceed 25 ml in volume.

A Bunsen burner.

Method:

About 120 g of the sample shall be distilled from a 250 ml distillation flask (e.g. Schedule No. F5, page 365) with adequate condensation, the distillate being collected in a small separating funnel. The distillation shall be stopped when water ceases to come over; the contents of the separating funnel shall be allowed to settle, the water run off and the oil layer shall be returned to the distillation flask when the latter has cooled to about 40°C. The contents of the flask shall be thoroughly mixed; 100 g of the mixture shall be weighed directly into the weighed distillation flask Schedule No. F4, and the apparatus assembled as described under "Apparatus" above (see Note i).

The distillation shall be started, using the naked flame of the burner, and continued at the rate of 5 ml \pm 0.5 per minute; if for any reason the distillation rate falls outside the specified limits at any time after the first 5 ml of distillate have collected and before the final specified temperature is reached, the test shall be discarded and another test started on a further portion of the sample. The specified distillation rate corresponds approximately to 90 drops per minute or three drops in each two seconds, but this must only be taken as a guide and graduated receivers are specified for the collection of the distillate in order that the rate in millilitres per minute may be kept under close observation.

The receiver shall be changed at each specified temperature (see under "Temperature Corrections" below) without stopping the distillation, except that the flame shall be extinguished

when the thermometer reaches the highest specified temperature and the final fraction shall include the oil which drains from the condenser within five minutes after the flame has been extinguished. The weight of each fraction shall be reported (see Note ii).

Should solids tend to deposit during the distillation, the condenser shall be warmed so that such solids are collected in the fraction with which they come over.

Temperature Corrections:

In the absence of other instructions, the boiling-points shall be corrected for the effect of barometric pressure according to the instructions on pages 64 to 69.

Notes:

(i) If the percentages of the distillates are required by volume, 100 ml of the dehydrated sample shall be used instead of the 100 g specified and in the absence of other instructions, the volumes of distillates shall be measured at the temperature at which the dehydrated sample was measured.

(ii) If a determination of the total phenols be required and the final specified temperature be below 350°C, the receiver shall be changed and the distillation restarted and continued to 350°C or the pitching-point (see page 63) if lower; this distillate shall also be weighed and all the distillates reserved for the phenols test described in Serial No. C.O. 4, below.

SERIAL No.: C.O. 4-38

Phenols

General:

The phenols are extracted by caustic soda and the soda solution is boiled to remove neutral oils and bases; the phenols are liberated by the addition of hydrochloric acid and their volume is measured.

Material for Test:

Unless the specification or other instructions require otherwise, the material for test shall consist of the whole of the distillate to 350°C (in the distillation test, Serial No. C.O. 3, page 270) or to the pitching-point if this be below 350°C.

Method:

The total volume of the material for test as described above shall be noted and the material, warmed if necessary until

completely liquid, shall be transferred to a stoppered separating funnel of appropriate capacity. Fifty millilitres (see Notes i and ii) of a 10 *per cent w/w* clear aqueous caustic soda solution shall be used to rinse the receivers from which the distillates were transferred and added to the latter in the separating funnel. If the combined distillates should contain naphthalene which tends to separate, both the distillate and the caustic soda solution shall be warmed just sufficiently to maintain the naphthalene in solution. The contents of the funnel shall be agitated vigorously for five minutes and allowed to stand.

After settlement and dilution of the upper layer with phenols-free naphtha if necessary to secure satisfactory separation, the alkaline layer shall be run into a beaker. The upper layer shall be agitated for five minutes with a further 25 ml of the soda solution and with further successive quantities each of 25 ml of the soda solution until all the phenols have been removed. This can be ascertained by *slightly* acidifying the last washings, using concentrated hydrochloric acid, and examining for separated phenols. After settlement, the alkaline layer shall in each case be added to the first soda washing. A large excess of soda must be avoided but some excess must be maintained; as a rough guide, it may be assumed that 25 ml of caustic soda solution of the strength indicated are sufficient to remove about 5 ml of phenols. During the washing processes, the contents of the funnel shall be kept liquid, immersing the funnel if necessary in warm water at between 40° and 70°C. (It may be necessary to reserve the washed distillate for examination for solid hydrocarbons according to the method described in Serial No. C.O. 5, page 274.)

The soda washings, including any acidified for the purpose of checking the washing procedure, shall be combined. The combined washings shall be alkaline; they shall be boiled vigorously for twenty minutes; fragments of porous or other suitable inert material may be used to prevent bumping. The soda washings shall be cooled to laboratory temperature and, if clear, shall be transferred direct to the phenols flask (Schedule No. V5, page 347—see Notes i and ii). If the solution be not clear, it shall be filtered through glass wool previously moistened with saturated aqueous sodium chloride solution; the filtrate shall be collected in the phenols flask; the glass wool shall be washed with 25 ml of saturated sodium chloride solution which shall be added to the filtered soda

washings. Methyl orange indicator solution shall be added followed by concentrated hydrochloric acid until the methyl orange just indicates distinct acidity after vigorous shaking together of the two layers. During the addition of the hydrochloric acid, the contents of the flask shall be kept cool by immersing the flask from time to time in cold water.

Just sufficient powdered sodium chloride shall be added to saturate the aqueous layer and leave a *few* particles undissolved; the phenols shall be brought into the graduated portion of the flask by adding saturated sodium chloride solution.

After adequate time for settlement, preferably overnight, the volume of phenols shall be read at the temperature at which the total volume of the material for test was measured and the percentage by volume of the phenols in the original oil or in the distillate shall be calculated and reported according to the requirements of the specification.

Notes:

(i) In the case of samples containing only small amounts of phenols, it is preferable to use half the specified volumes of caustic soda solution for the successive washings and to collect the soda washings (after filtration through the glass wool if necessary) in the alternative phenols flask Schedule No. V5a, page 347.

(ii) If it be expected that the material for test contains 25 *per cent* or more of phenols (as for example in certain samples of cresylic creosote), the volumes of caustic soda solution shall be appropriately increased. The boiled soda washings (after filtration through the glass wool if necessary) shall be divided about evenly between two phenols flasks, the subsequent specified procedure being carried out on the contents of each flask and the volumes of phenols recovered being totalled.

SERIAL No.: C.O. 5-38

Solid Hydrocarbons

It is to be understood that this determination is only in respect of the hydrocarbons deposited from solution at 15.5°C (see Note i).

Material for Test:

The following test will usually be performed on the sample as received. Occasionally the determination is required on material freed from phenols in which case the distillate after

washing with caustic soda solution in the test for phenols (Serial No. C.O. 4, page 272) shall be used.

Method:

A portion of the sample shall be warmed if necessary until liquid and shall be well shaken. Fifty grams shall be cooled to $15.5^{\circ}\text{C} \pm 0.5$ Centigrade degree and, after seeding with a small crystal of naphthalene if no crystalline material has appeared by then in the cooled sample, shall be maintained within those limits of temperature for four hours with occasional stirring.

The material shall then be transferred to a cold Buchner funnel fitted with a rapid filter paper, and as much oil as possible removed rapidly by a filter pump. The residue shall be placed between sheets of blotting paper and submitted to pressure in a hand screw-press. The oily margins of the cake shall be removed and separately pressed.

The operations, starting with the transfer to the Buchner funnel to the preliminary pressing, shall be conducted with the utmost rapidity.

The pressed residue shall be weighed and the weight in grams multiplied by 2 to give the percentage by weight of solid hydrocarbons deposited at 15.5°C from the sample (see Note ii).

Notes:

(i) The above method has been formulated for application in the most usual circumstances, that is to say where the amount of solid hydrocarbons at 15.5°C is required. The procedure is obviously easily adaptable if the amount at any other temperature be required, but *an indication of any deviation from the standard temperature of 15.5°C shall be recorded in the analyst's report.*

(ii) The result will include any insoluble matter which may be present in the material for test.

SERIAL No.: C.O. 6-38

Matter Insoluble in Toluole

Method:

Twenty grams or more of the well mixed sample shall be weighed into a 200 ml beaker and dissolved by warming on a water-bath, in about four times its volume of 90's toluole

(B.S. 805—1938, Part C), the beaker being loosely covered. The solution shall be filtered, either (a) through balanced filter papers (e.g. Whatman No. 5) about 150 mm in diameter, or (b) through a weighed Gooch crucible prepared with asbestos and used under vacuum.

If filter papers are used, they must be heated to a temperature between 95° and 110°C and reduced to equal weight by removing the apex of the heavier paper; the uncut paper is used inside the originally heavier paper. The separated material is retained by the inner paper, but both of them are equally subjected to any action exerted by the toluole.

Any insoluble residue shall be transferred to the filter by means of more of the toluole and the filter and contents washed with the solvent until a few drops of the filtrate yield no residue on evaporation. The residue shall be dried in an oven at a temperature between 95° and 110°C until it is of constant weight. The percentage by weight of insoluble matter in the sample shall be calculated and reported.

SERIAL No.: C.O. 7-38

Ash

Method:

About 200 g of the sample shall be weighed accurately into a weighed distillation flask and distilled until only a small residue remains, avoiding the formation of coke. The flask and contents shall be weighed, about half the well mixed contents shall be transferred to a weighed porcelain or silica crucible and the crucible and contents weighed.

The crucible shall be heated cautiously to expel volatile matter, avoiding ebullition, and finally ignited until all combustible material is burnt. The crucible shall be allowed to cool and shall be weighed. The percentage by weight of ash in the sample shall be calculated and reported.

SERIAL No.: C.O. 8-38

Coke Residue

(After P. H. Conradson, *8th Int. Con. app. Chem.*, 1912, 1, Section I, p. 131.)

General:

The following test is a means of determining the amount of coke residue left on evaporation under specified conditions and

is intended to throw some light on the relative coke-forming tendency of an oil. The results of the test must be considered in connexion with other tests and with the use for which the oil is intended.

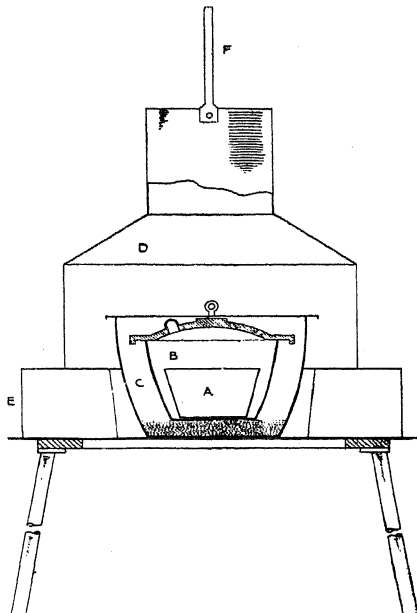


Fig. 26.—Coke Residue on Evaporation of Creosote Oil—Assembled Apparatus.

Apparatus:

The apparatus shall be as shewn in Fig. 26, above, and as described below:—

A silica or porcelain crucible (A) of squat shape, glazed throughout and having a capacity of $30\text{ ml} \pm 1$ and a rim diameter (external) of $47\text{ mm} \pm 1$.

A Skidmore iron crucible (B) flanged and ringed, having a capacity of $74\text{ ml} \pm 8$; the flange shall have an internal diameter of $55\text{ mm} \pm 2$ and an external diameter of $63\text{ mm} \pm 3$. The crucible shall be $38\text{ mm} \pm 1$ in height, and shall be supplied with a cover without delivery tubes and having the vertical

opening closed. The horizontal opening (about 6.5 mm in diameter) shall be kept clean. The flat bottom of the crucible shall have an external diameter of 31 mm \pm 1.

A spun sheet-iron crucible (C) 80 mm \pm 2 in external diameter at the top, 59 mm \pm 1 in height and about 0.8 mm in thickness. The crucible shall be provided with a cover. Sufficient (about 25 ml bulk volume) dry sand shall be placed in this crucible to bring the cover on the Skidmore crucible nearly to the top of the sheet-iron crucible.

A triangle of bare No. 15 I.W.G. nichrome wire having an opening small enough to support the bottom of the sheet-iron crucible at the same level as the bottom of an asbestos block or a hollow sheet-metal box (E) (see below).

A circular sheet-iron hood (D) 125 mm \pm 5 in diameter, the height of the lower perpendicular side being 51 mm \pm 1; the hood shall be provided at the top with a chimney 55 mm \pm 5 in height, 53 mm \pm 3 in internal diameter and attached to the lowest part of the hood by a cone shaped member; the total height of the complete hood shall be between 125 and 130 mm. The hood may be made from a single piece of metal provided all the foregoing dimensions are observed. A bridge (F), made of No. 10 I.W.G. iron or nichrome wire and having a height of 50 mm above the top of the chimney, forms a guide for the height of the flame above the chimney.

An asbestos block or hollow sheet-metal box (E) 160 mm \pm 10 in diameter if round or on a side if square, 35 mm \pm 3 in thickness and provided with a metal lined, inverted cone shaped opening through the centre; this opening shall be 83 mm in diameter at the bottom, and 89 mm in diameter at the top.

A Meker burner having a diameter of 24 mm at the top; the flame diameter is 25 mm.

Material for Test:

The determination shall be carried out on the dried sample, and the sample as received shall therefore be treated with plaster of Paris as follows:—

About 100 g of finely powdered plaster of Paris (calcium sulphate hemihydrate) shall be placed in a dry wide mouthed stoppered glass bottle having a capacity of about 450 ml and an external diameter of from 50 to 60 mm. About 250 ml of the sample shall be introduced and the stopper of the bottle secured in position by tying. The bottle shall be well shaken

and placed vertically in a vessel of water, the level of the water reaching to about one third of the height of the oil. The water shall be warmed to 50°C and maintained at this temperature for a period of seven hours, the bottle and contents being well shaken at intervals of about an hour. At the end of this treatment the bottle shall be opened and the required quantity of the dried sample shall be decanted directly into the porcelain or silica crucible as described under "Method" below.

Method:

Two glass beads about 2.5 mm in diameter shall be placed in and included with the weight of the weighed porcelain or silica crucible; 10 g of the material for test (see above) shall be weighed to within 5 mg into the crucible, which shall then be placed in the centre of the Skidmore crucible. The sand in the sheet-iron crucible shall be levelled and the Skidmore crucible set on it and exactly central with the iron crucible. The covers shall be placed on the Skidmore and the iron crucibles, that on the latter fitting loosely to allow free exit for the vapours from the sample.

The nichrome wire triangle shall be placed on a suitable stand or ring and the asbestos block or hollow sheet-metal box shall be placed on the triangle. The sheet-iron crucible shall be placed on the triangle and central with the block or box, and the sheet-iron hood shall be placed central on the latter in order to distribute the heat uniformly during the heating process (see Fig. 26, page 277).

Heat shall be applied with the full flame of the Meker burner so that the pre-ignition period will be 10 minutes ± 1.5 (a shorter time may start the distillation so rapidly as to cause foaming or too high a flame above the chimney). When smoke appears above the chimney, the burner shall be immediately moved or tilted so that the gas flame plays on the sides of the crucible and the vapours are ignited. The source of heat shall then be temporarily removed and replaced after adjusting the flame so that the ignited vapours burn uniformly with the flame above the chimney but not above the wire bridge. The supply of heat may be increased if necessary when the flame does not show above the chimney. The period of burning the vapours shall be 13 minutes ± 1 .

When the vapours cease to burn and no further smoke can be observed, the burner shall be re-adjusted and heat supplied

as at the beginning so that the bottom and lower part of the sheet-iron crucible become a cherry red and are so maintained for exactly 7 minutes. The total period of heating shall be 30 minutes ± 2 (see Note i).

The burner shall be removed and the apparatus allowed to cool until no smoke appears (about 15 minutes), when the cover of the Skidmore crucible shall be removed. The porcelain or silica crucible shall be taken out with heated tongs and placed in a desiccator where it shall be allowed to cool. It shall then be weighed and the percentage by weight of coke residue shall be calculated and reported on the dried sample.

Limits of Accuracy:

Tests shall be made in duplicate and repeated if necessary until the results of two successive tests fall within ± 10 per cent of the average of the two results.

Notes:

(i) The limit of 30 minutes ± 2 for the total period of heating constitutes an additional limitation on the tolerances for the pre-ignition and burning periods, but there should be no difficulty in carrying out the test exactly as directed with the burner specified, using town gas (about 500 B.Th.U.) and with the top of the burner about 50 mm below the bottom of the crucible. The time periods shall be observed even if any other burner or gas must of necessity be used.

(ii) The above method is, apart from editorial unification with the present publication, identical with the method Serial Designation G.O. 9 in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 31.

SERIAL No.: C.O. 9-38

Fluidity

General:

Creosote oil specifications generally require that the oil shall remain completely fluid for a specified period of time at a specified temperature. On occasion, the temperature at which soluble solid matter first separates on cooling is required. The following method consists of two parts, each providing for one of the foregoing sets of circumstances.

Method:**PART A. EXAMINATION FOR FLUIDITY AT A SPECIFIED TEMPERATURE**

About 50 g of the thoroughly mixed sample shall be weighed into a 100 ml conical flask. A standard thermometer, Schedule No. T1a (-10° to $+20^{\circ}\text{C}$) or T5a (15° to 45°C) according to the circumstances, shall be fitted by means of a cork in the neck of the flask, with the bulb of the thermometer immersed in the oil. The flask and contents shall be brought, with occasional gentle shaking, to a temperature just sufficient to secure the complete fluidity of the sample.

The flask and contents shall be cooled slowly to the temperature of test with similar occasional gentle shaking to induce crystallization. The temperature of test shall be maintained by means of a thermostat bath for two hours or for such other period of time as may be specified. The flask shall then be withdrawn from the bath, rotated slowly in a horizontal position and examined for the separation of soluble solid matter which if present will be seen adhering to the side of the flask or floating on the surface of the oil.

If there be any doubt as to the completeness or otherwise of fluidity, the contents of the flask shall be rapidly filtered through a Buchner funnel fitted with a rapid filter paper and operating under vacuum. The funnel shall have been previously brought to about the temperature of test, either by placing it in an air bath or by dipping it in a creosote oil bath at that temperature, according to convenience. The paper shall be examined for any soluble solid matter which may have separated from the sample.

PART B. DETERMINATION OF TEMPERATURE OF DEPOSITION OF SOLUBLE SOLID MATTER

About 50 g of the thoroughly mixed sample shall be weighed into a 100 ml conical flask. A standard thermometer, Schedule No. T1a (-10° to $+20^{\circ}\text{C}$) or T5a (15° to 45°C) according as the deposition temperature is expected to be below or above 20°C , shall be fitted by means of a cork in the neck of the flask, with the bulb of the thermometer immersed in the oil. The flask and contents shall be brought, with occasional gentle shaking, to a temperature just sufficient to secure the complete fluidity of the sample.

The flask and contents shall be cooled by immersion in a bath maintained at ten Centigrade degrees below the expected

deposition-point. The flask shall be frequently rotated in a horizontal position and withdrawn at intervals for examination for the separation of soluble solid matter which, when it appears, will be seen adhering to the side of the flask or floating on the surface of the oil. The temperature at which soluble solid matter first separates shall be reported.

SERIAL No.: C.O. 10-38

Flash-point

Apparatus:

A Pensky-Martens closed tester conforming to the specification of the Institute of Petroleum (see "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 99). The instrument shall be certified and marked by the National Physical Laboratory as complying with the dimensions specified.

Material for Test:

The flash-point is affected by the presence of water and the sample shall therefore be treated with plaster of Paris as follows:—

About 100 g of finely powdered plaster of Paris (calcium sulphate hemihydrate) shall be placed in a dry wide mouthed stoppered glass bottle having a capacity of about 450 ml and an external diameter of from 50 to 60 mm. About 250 ml of the sample shall be introduced and the stopper of the bottle secured in position by tying. The bottle shall be well shaken and placed vertically in a vessel of water, the level of the water reaching to about one third of the height of the oil. The water shall be warmed to 50°C and maintained at this temperature for a period of seven hours, the bottle and contents being well shaken at intervals of about an hour. At the end of this treatment the bottle and its contents shall be allowed to cool to laboratory temperature. The bottle shall be opened, sufficient of the dried and settled sample decanted into the testing cup of the Pensky-Martens flash-point apparatus and the flash-point determined as described below.

Method:

All parts of the cup of the instrument and its accessories shall be thoroughly clean and dry before starting the test, avoiding particularly the presence of any inflammable products used to clean the apparatus after a previous test.

The cup shall be filled with the material for test (see above) to the level indicated by the filling mark. The lid shall be placed on the cup and the latter set in the stove, the locating devices being properly engaged. The "P.M.Low" thermometer specified for the instrument shall be inserted. The test flame shall be adjusted so that it is of the size of a bead 0.16 inch (4 mm) in diameter.

Heat shall be applied at such a rate that the temperature increases not less than 9 and not more than 11 Fahrenheit degrees per minute. The stirrer shall be turned at a rate of about 60 revolutions per minute.

Application of the test flame shall be made at each temperature reading which is a multiple of 2 Fahrenheit degrees up to 220°F. The first application of the test flame shall be made at a temperature at least 50 Fahrenheit degrees below the expected flash-point. Application of the test flame shall be made by operating the device controlling the shutter and test flame burner so that the flame is lowered in one half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

The flash-point is the temperature at the time of that flame application which causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo which sometimes surrounds the test flame during the applications preceding the one which causes the actual flash.

The barometric pressure shall be observed and recorded; corrections shall be applied to the observed flash-point by adding or subtracting 0.63 Fahrenheit degree for each 10 mm by which the barometric pressure is below or above 760 mm as the case may be.

Note:

The above method is, apart from editorial unification with the present publication, identical with the method Serial Designation G.O. 7 in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 99.

SERIAL No.: C.O. 11-38

Viscosity

Apparatus:

A Redwood No. 1 viscometer conforming to the specification of the Institute of Petroleum (see "Standard Methods for

Testing Petroleum and its Products," 1935 edition, page 161). The instrument shall have been submitted to the National Physical Laboratory for certification; its compliance with the specification as to general construction and dimensions will thereby be checked; it will also be subjected to a flow test in comparison with a standard reference viscometer at the Laboratory and must agree within ± 1.5 per cent of the time of flow of the reference viscometer. Instruments complying with the specification and being of the required accuracy will be certified by the N.P.L.

A 50 ml Kohrausch flask complying with B.S. 615—1936.

Method:

The oil cup of the viscometer shall be cleaned with a suitable solvent (ether, pure benzole, petroleum spirit, etc.) and thoroughly dried to remove all traces of the solvent employed. If any material be used for rubbing the interior of the oil cup or for cleaning the jet, it shall not be such as will disintegrate in use, leaving particles which may interfere with the free flow of the sample through the jet. The apparatus shall be set up and levelled.

About 200 ml of the sample shall be heated in a separate vessel to within a few degrees of 100°F. Either water or oil may be used in the heating bath; the level of the heating liquid shall be at least 10 mm below the rim of the cup. The bath of the viscometer shall be heated to a few degrees above 100°F and the oil poured into the oil cup through a filter of fine mesh gauze (at least 100 mesh per inch). The temperature of the bath shall be adjusted until the temperature of the oil in the cup is maintained at 100°F, the stirrer in the bath being gently rotated at frequent intervals or (preferably) continuously. The oil shall be gently stirred during the preliminary period, but shall not be stirred during the actual test. When the oil has reached a steady temperature of 100°F, the adjustment of the oil level shall be made by allowing the oil to flow out until the surface of the oil touches the filling-point. The oil cup cover shall be slightly warmed and placed in position. The standard flask shall be placed centrally below the jet, the top of the neck of the flask being adjusted to within a few millimetres of the under side of the jet. The flask shall not be insulated in any way. The valve shall be lifted and a stop watch started simultaneously. The watch shall be stopped when the oil reaches the graduation mark on the flask and the reading of the oil cup thermometer shall

be reported. The interval in seconds between the two readings of the stop watch shall be reported as the viscosity of the sample (see Note i).

Notes:

(i) The method as described provides for the determination of the viscosity at 100°F, which is the temperature usually specified. *If there be any variation from the standard method by the use of any other temperature of determination, that fact shall be recorded in the analyst's report.*

(ii) The above method is, apart from editorial unification with the present publication and modification of general instructions so that they apply particularly to the examination of creosote oil, identical with the method Serial Designation L.O. 8 in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 161.

SERIAL No.: C.O. 12-38

Calorific Value

General:

The calorific value as determined below is the gross calorific value of the oil, expressed as 15°C calories under conditions of constant volume for the products of combustion; this value may differ from that at constant pressure by 1 in 1,000. The net value has no exact meaning apart from the conditions under which the oil is burnt.

The 15°C calorie is 1 in 1,000 greater than the 20°C calorie which is sometimes used. The following data may be used for correcting determinations made at temperatures varying from 15°C for the variation of the specific heat of water, which is taken as 1,000 at 15°C:—

15°C Calories

<i>Mean temperature of chief period ($t_o + t_n$)/2 °C</i>	<i>Correction for each degree rise of temperature Centigrade degree</i>
Below 7.5	calculate
7.5 to 8.5	add 0.003
8.5 to 11.5	add 0.002
11.5 to 13.5	add 0.001
13.5 to 17.5	no correction
17.5 to 22.5	subtract 0.001
22.5 to 27.5	subtract 0.002

If the water equivalent of the calorimeter has been determined at different temperatures, the above correction is unnecessary.

The definitions of and relations between various recognized heat units are shewn on page 384.

Apparatus, etc.:

(a) *Combustion Bomb*: A high pressure bomb having an inner surface which is not attacked by acids to an extent which would interfere with the accuracy of the results. The capacity of the bomb shall be such that when closed and filled with oxygen at the working pressure, it will contain at least two and a half times the amount of oxygen required for combustion of the portion of the sample taken for test.

(b) *Calorimeter Jacket*: The calorimeter shall be provided with an outer jacket containing sufficient water to provide a constant temperature enclosure during the time of the experiment. The water capacity of the outer jacket shall be at least five times that of the total water equivalent of the calorimeter charged with water. A uniform temperature of the water jacket may be secured by stirring.

(c) *Stirring of the Calorimeter Water*: The water in the calorimeter shall be efficiently stirred; the rate of stirring shall be constant and such that the temperature readings plotted against the time shall lie on a smooth curve.

(d) *Thermometers*: The thermometers used shall have been certified by the National Physical Laboratory. They shall be graduated to 0.01 Centigrade degree and used with certified corrections which shall be given to within 0.005 Centigrade degree, and, if the reliability of the thermometer permits, to 0.002 Centigrade degree.

If a thermometer of the Beckmann type be used and the certificate of testing does not shew the changing value of the degree due to the variable volume of mercury in the bulb, determinations of the water equivalent shall be made at various temperatures say, when the zero of the thermometer corresponds to 10°, 15°, 20°C (about), and the appropriate value used when calorimetric determinations are made.

(e) *Oxygen*: The oxygen used for combustion shall be available at a pressure of not less than 25 atmospheres, and shall be free from combustible matter, including hydrogen (small percentages of which may be present in electrolytic oxygen).

(f) *Firing Wire*: The sample in the bomb shall be electrically ignited by means of platinum wire about 0.08 mm in diameter.

Water Equivalent of Apparatus:

The water equivalent of the apparatus shall be determined by burning a known weight of pure benzoic acid (about 1.2 g is a convenient quantity) in the bomb in the manner described under "Method" below. The calorific value of benzoic acid is 6,324 calories (15°C) per gram weight in air. The benzoic acid shall be carefully dried either by being kept in a desiccator over sulphuric acid for a prolonged period or by being heated to not more than five Centigrade degrees above its melting-point (121.7°C). The dry benzoic acid shall be kept in a desiccator except when being weighed or pressed into briquettes. It shall be burnt in the form of a briquette or as solidified after fusion. It may conveniently be fused in the weighed crucible in which it is to be burnt.

Method:

It is desirable that the temperature of the working room should vary as little as possible and be near 15°C.

About 0.8 g of the sample shall be weighed accurately into the crucible of the calorimeter.

A piece of the platinum firing wire shall be stretched across the pole pieces of the bomb. One end of a piece of sewing cotton shall be tied round the wire, the crucible placed in position and the loose end of the cotton arranged so that it is in contact with the oil. The same amount of cotton shall be used in each test.

Ten grams of distilled water shall be introduced into the body of the bomb; the bomb shall be reassembled and screwed home with the fingers, finally tightening with a spanner if necessary, but avoiding excessive pressure. Care shall be taken that none of the creosote oil is lost from the crucible during the assembly of the apparatus.

The bomb shall be charged slowly with oxygen to 25 atmospheres pressure, without displacing its original air content. The valve shall be closed effectively, using as little pressure as possible, and the bomb disconnected.

A quantity of water at laboratory temperature and sufficient to submerge the cover of the bomb (leaving the terminals projecting) shall be weighed in the calorimeter vessel, choosing a weight of water which with the 10 g in the bomb and the

water equivalent of the apparatus will bring the total water value to a convenient figure. To ensure a peak in the rise of temperature due to the combustion of the oil, the water in the calorimeter vessel shall be not more than two Centigrade degrees below the temperature of the air chamber of the water jacket. The calorimeter vessel shall be transferred to the water jacket and the bomb lowered carefully into the calorimeter vessel. The bomb shall be examined to ensure that it is gas-tight and shall be connected to a battery or a suitable transformer giving 4 to 6 volts, through an open key, for the subsequent firing of the charge. The stirrer shall be adjusted, the thermometer and covers placed in position and the stirring mechanism started; stirring shall be continuous during the remainder of the test.

After an interval of not less than five minutes, the temperature shall be read accurately, the temperature readings being noted for five minutes at equal intervals of one minute, tapping the thermometer lightly at the level of the top of the mercury column during ten seconds prior to each reading. At the end of the fifth minute the battery circuit shall be *momentarily* closed to fire the charge and the observations of the temperature shall be continued at intervals of one minute.

In the chief period, which extends from the instant of firing until the time after which the rate of change of temperature again becomes constant, the earlier readings shall be recorded to the nearest 0.01 Centigrade degree, but the later readings to the nearest 0.002 Centigrade degree. The readings shall be continued at one minute intervals until the maximum temperature is passed and the fall of temperature, which at first is very slow, has assumed a steady rate for not less than five minutes as shewn by consecutive readings.

The bomb shall be removed from the calorimeter and, after the lapse of half an hour from the time of firing to allow the acid mist to settle, the pressure shall be released by opening the valve (if the total sulphur content of the sample be required, the procedure for cooling and opening the bomb as described in Serial No. C.O. 13, page 293, shall be followed). The combustion must be complete as shewn by the absence of any sooty deposit within the bomb.

Corrections:

(a) *Cooling Correction:* The cooling correction to due Regnault and to Pfaundler has the form:—

$$\text{Correction} = nv + \frac{(v' - v)}{(t' - t)} \left\{ \sum_1^{n-1} (t) + \frac{1}{2}(t_0 + t_n) - nt \right\} \\ nv + kT$$

where

n = the number of minutes in the chief period;

v = the rate of fall of temperature per minute in the preliminary period (if the temperature in this period be rising, v is negative);

v' = the rate of fall of temperature per minute in the after period;

t = the average temperature during the preliminary period;

t' = the average temperature during the after period;

$\sum_1^{n-1} (t)$ = sum of the readings ($t_1, t_2, t_3, \dots, t_{n-1}$) during the chief period;

$\frac{1}{2}(t_0 + t_n)$ = mean of firing temperature t_0 and first temperature t_n after which the rate of change is constant; and

$k = \frac{(v' - v)}{(t' - t)}$ and is the "cooling constant" of the calorimeter.

For an instrument with a water equivalent of about 2,500 g, the value of k should not exceed 0.0025, if the heat insulation be adequate.

From the nature of the formula it is seen that the temperature difference $t_n - t_0$, to which this correction is added to obtain the corrected rise, is not the maximum observed rise. In fact, within reasonable limits, the corrected rise should be the same, however far along the cooling curve t_n is taken, provided the period of uniform fall of temperature has been reached.

The use of a blank form, with spaces for insertion of the various readings, greatly facilitates the application of the Regnault-Pfaundler correction and reduces the liability to errors of calculation (see table on page 292). It has been found that experience with this correction may lead to a shortened expression with the same accuracy, but this is applicable only to a given set of apparatus and conditions.

(b) *Correction for Cotton Thread:* The correction for the cotton thread used for firing the charge is calculated from the weight of a known length of the thread dried at 110°C and the calorific value of cellulose, viz. 4,140 calories per gram.

(c) *Correction for Sulphur and Nitrogen:* The heat evolved by the combustion of the creosote oil in the bomb with oxygen includes the heat due to the formation and solution of sulphuric acid from any sulphur in the creosote oil, and that due to the formation and solution of nitric acid from the nitrogen of the air left in the bomb and from some of the nitrogen in the creosote oil. The corrections to be made shall be obtained as follows:—

(i) *If the total sulphur has been determined independently* (see Serial No. C.O. 13, page 293), this value may be used to derive the necessary correction, the nitric acid being determined by deducting from the total acidity of the bomb washings that due to the sulphuric acid formed during the combustion, as follows:—

The contents of the bomb shall be washed into a hard glass beaker with distilled water and the valve and capsule similarly washed into the beaker; any deposit of lead sulphate on the washer shall also be included in the washings. A measured excess of 0.1N sodium carbonate solution (50 ml will usually suffice) shall be added and the contents of the beaker shall be boiled down to about 10 ml; they shall be diluted, filtered and washed. The cold filtrate shall be titrated with 0.1N hydrochloric acid, using two or three drops of methyl orange solution as indicator.

If the weight of sulphur in the creosote oil burnt be w grams, the corresponding volume of 0.1N sulphuric acid is $w/0.0016$ ml. If v_1 ml of 0.1N hydrochloric acid are used in the titration, the total acidity due to nitric and sulphuric acids together is equivalent to $(50 - v_1)$ ml of 0.1N acid and the amount of nitric acid present is equivalent to $50 - v_1 - w/0.0016$ ml of 0.1N nitric acid.

To correct the calorific value as determined in the bomb calorimeter to that which would be obtained by the complete combustion of the creosote oil in air, 1.43 calories shall be deducted for each millilitre of 0.1N nitric acid produced, and 22.5 calories for each 0.01 g of sulphur, from the total number of calories developed.

(ii) *If the sulphur has not been determined independently*, the solution which has been titrated under (i) above may be

acidified with hydrochloric acid and the sulphur in it determined as described under Serial No. C.O. 13, page 293.

(iii) *Alternatively, when it is unnecessary to determine the sulphur by the standard method (in order to report the sulphur content as such), the following volumetric method of determining the nitric and sulphuric acids produced may be used, provided a bomb as prescribed, the lining of which is not attacked by the acid produced in the combustion, has been used:—*

The contents of the bomb having been washed into the beaker as described above, the dissolved carbon dioxide shall be removed by boiling and the solution titrated while hot with 0.1N barium hydroxide solution, using two or three drops of phenolphthalein solution as indicator. Twenty millilitres of 0.1N sodium carbonate solution shall be added, the mixture warmed and filtered and the residue washed. The washings shall be added to the filtrate, which shall be titrated when cold with 0.1N hydrochloric acid, using two or three drops of methyl orange solution as indicator, the phenolphthalein colour change being ignored.

If v_2 ml of 0.1N barium hydroxide solution are required for the acidimetric titration with phenolphthalein and v_3 ml of 0.1N hydrochloric acid are required to neutralize the residual alkalinity after precipitation and removal of barium carbonate, then there were present in the washings from the bomb the equivalents of $v_2 - (20 - v_3)$ ml of 0.1N sulphuric acid and $(20 - v_3)$ ml of 0.1N nitric acid.

EXAMPLE

The following example illustrates the method and calculations:—

Capsule + creosote oil	=	7.6357 g
Capsule	=	6.7191 g
Creosote oil	=	0.9166 g
Water	=	2,367 g
Total water value	=	2,800 g
Final temperature of water	=	16.5°C

Preliminary period		Chief period		After period	
Time	Temperature	Time	Temperature	Time	Temperature
0	1.129	6 (t_1)	1.87	12 (t_7)	4.081
1	1.133	7 (t_2)	3.17	13	4.078
2	1.137	8 (t_3)	3.805	14	4.074
3	1.141	9 (t_4)	4.002	15	4.071
4	1.144	10 (t_5)	4.068	16	4.068
5 (t_0)	1.148	11 (t_6)	4.080	17	4.065
$t = 1.139$		$\sum_{i=1}^{n-1}(t) = 20.995$		$t' = 4.073$	
$v = -0.0038$		$\frac{1}{2}(t_0 + t_n) = 2.615$		$v' = 0.0032$	
$n = 7$		23.610		$k = \frac{v' - v}{t' - t} = 0.00239$	
$nt = 7.973$		$-nt = -7.973$			
$nv = -0.0266$		$T = 15.637$		$kT = 0.0374$	
Cooling correction = $nv + kT$					
= $-0.0266 + 0.0374$					
= $+0.011$ Centigrade degree					

0.1N sodium carbonate solution

= 25 ml

0.1N hydrochloric acid (v_1)

= 14.8 ml

Barium sulphate

= 0.0541 g

Combustible sulphur (w) = 0.0541×0.1373

= 0.0074 g

Correction for nitric acid = $(25 - v_1 - w/0.0016) \times 1.43$

$$= \left\{ 25 - 14.8 - \frac{0.0074}{0.0016} \right\} \times 1.43 = 8 \text{ calories}$$

Correction for sulphuric acid = 0.0074×2250

= 17 calories

Uncorrected temperature rise ($t_n - t_0$)

Cent. degrees

Cooling correction

= 2.933

Calibration correction

= +0.011

Correction to 15°C calories

= -0.003

Corrected rise

= nil

Heat liberated = $2.941 \times 2,800$

= 2.941

Correction for thread -20

= 8,235 calories

Correction for nitric acid - 8

= -45 calories

Correction for sulphuric acid -17

Heat from 0.9166 g of creosote oil

= 8,190 calories

Heat from one gram of creosote oil

= 8,935 calories

Calorific value

= 8,935 calories

or, multiplying by 1.8,

$$= 16,083 \frac{\text{B.Th.U.}}{\text{per gram}}$$

Such a result should be rounded off to 16,080 B.Th.U. per lb.

Limits of Accuracy:

The results of two consecutive tests should fall within thirty units of each other and the mean of the two readings should be within ± 0.2 per cent of the true value.

SERIAL No.: C.O. 13-38

Total Sulphur

Apparatus:

A high pressure combustion bomb and accessory equipment as described in Serial No. C.O. 12, page 285, for the determination of the calorific value. The additional items of apparatus required will depend on whether the total sulphur content is alone required or the calorific value also.

Reagent:

Barium chloride solution: An aqueous solution containing 10 grams of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in each 100 ml of solution.

This solution, the distilled water and all the reagents used in the test shall if possible be free from sulphur, but when it is necessary to employ reagents which are not free from sulphur, a blank test shall be carried out and an appropriate correction made.

Method:

The first part of the determination, involving the combustion of a portion of the sample, shall be carried out in the bomb calorimeter, the method of operation being as described in Serial No. C.O. 12, page 285. In many cases the two determinations will be made in effect simultaneously, the sulphur being determined as below and used additionally in connexion with the correction to be made therefor in the determination of the calorific value. If, however, the sulphur content be alone required, certain operations in the test for calorific value may obviously be omitted.

The bomb shall be allowed to cool and stand for at least ten minutes; the valve shall be opened, allowing the gas to escape at an approximately even rate so that the pressure is reduced to atmospheric in not less than one minute. The bomb shall be opened and all parts of its interior, including the oil cup, rinsed with a fine jet of distilled water, using in all not more than 350 ml. All the washings shall be collected in a beaker, care being taken not to lose any of the liquid contents of the bomb by splashing or otherwise. The washings shall be filtered through a washed qualitative filter paper and the filter paper washed thoroughly. Two millilitres of concentrated hydrochloric acid and 10 ml of saturated bromine

water shall be added to the filtrate and the solution evaporated to about 75 ml on a steam bath or hot plate. Ten millilitres of the hot barium chloride solution shall be added to the solution in a fine stream or by drops, stirring during the addition and for two minutes thereafter. The solution shall be allowed to stand overnight, or shall be kept hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for another hour. The supernatant liquid shall be filtered through an ashless quantitative filter paper and the precipitate washed with water, first by decantation, then on the filter, until the filtrate is free from chloride. The paper and precipitate shall be ignited and weighed in the usual way.

The percentage by weight of sulphur in the sample is—

$$13.74 \frac{W_2}{W_1}$$

where

W_1 = the weight of sample burnt; and

W_2 = the weight of barium sulphate obtained.

Note:

The above method is, apart from editorial unification with the present publication, identical with the method Serial Designation G.O. 4 in "Standard Methods for Testing Petroleum and its Products," 1935 edition, page 146.

SERIAL No.: C.O. 14-38

Creosote Oil—Distillation

(A.W.P.A. Method)

The method for the distillation of creosote oil as standardized by the American Wood Preservers' Association is frequently used in British laboratories, and the Committee has obtained from that Association permission to include a facsimile reproduction of the method in the present publication (see pages 295 to 298). The method is the same as that of the American Railway Engineering Association and that of the American Society for Testing Materials.

FACSIMILE OF U.S. CUSTOMS DISTILLATION METHOD FOR CREOSOTE

American Wood-Preservers' Association
Manual of Recommended Practice

11e

STANDARD DISTILLATION OF CREOSOTE

1. **Scope.** This method of test is suitable for all types and grades of creosote and mixtures of creosote with tars and oils used for timber preservation.

I. Apparatus

2. **Flask.** The distillation flask, Fig. 1, shall be a side neck distilling flask, having the following dimensions:

Diameter of bulb (outside).....	86 mm.	±1.5 mm
Diameter of neck (inside).....	22 "	±1.0 "
Diameter of tubulature (inside).....	10 "	±0.5 "
Height of flask (outside).....	131 "	±1.5 "
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature inside.....	93 "	±1.5 "
Length of tubulature.....	220 "	±5.0 "
Angle of tubulature.....	73 deg.	± 2 deg.
Thickness of tubulature wall.....	1.0 to 1.5 mm.	

3. **Condenser Tube.** The condenser tube shall be a suitable form of tapered glass tubing of the following dimensions:

Diameter of small end.....	12.5 mm.	permissible variation... ± 1.5 mm.
Diameter of large end.....	28.5 "	permissible variation... ± 3.0 "
Length.....	360.0 "	permissible variation... ± 4.0 "
Length of tapered part.....	100.0 "	permissible variation... ± 5.0 "

4. **Shield.** A galvanized-iron shield, lined with one-eighth inch asbestos, of the form and dimensions shown in Figure 2 shall be used to protect the flask from air currents and to prevent radiation. The cover (top) shall be of transite board made in two parts, or it may be of galvanized-iron lined with one-eighth inch asbestos.

5. **Receivers.** The distillates shall be collected in tared Erlenmeyer flasks having a capacity of 50 or 125 ml.

6. **Thermometer.** The thermometer shall conform to the following requirements. These specifications cover a total immersion thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the range being from 0 to 400° C. or 30 to 760° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and Subdivision: 0 to 400° C. in 1° C. or 30 to 760° F. in 2° F.

Total Length: 378 to 384 mm. (14.88 to 15.12 in.).

Stem: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

Bulb: Corning normal or equally suitable thermometric glass.

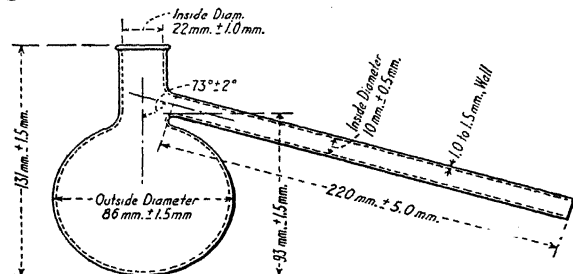


Fig. 1.—Distillation Flask

STANDARD METHODS

Length, 10 to 15 mm. (0.39 to 0.59 in.).

Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

Distance to 0° C. or 32° F. Line from Bottom of Bulb: 25 to 35 mm. (0.98 to 1.38 in.).

Distance to 400° C. or 752° F. Line from Top of Thermometer: 30 to 45 mm. (1.18 to 1.77 in.).

Filling above Mercury: Nitrogen gas.

Top Finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: Total.

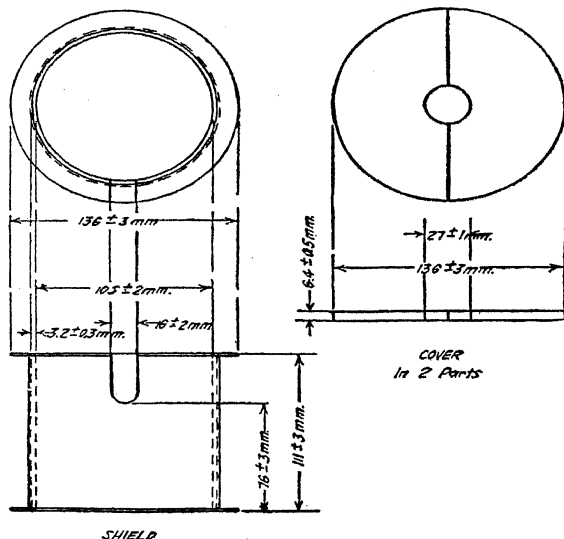
Special Marking: "A.S.T.M. High Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

Scale Error: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.

Test for Permanency of Range: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable



Flanged Open-End Cylinder
Made of 22 gage Galvanized Iron
with $\frac{1}{8}$ " Asbestos Lining Riveted to Metal

Fig. 2

SCALE 8 in = 1 ft.

case on which shall appear the marking: "A. S. T. M. High Distillation, 0 to 400° C." or "A. S. T. M. High Distillation, 30 to 760° F." according to the type of thermometer.

Note.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gauge.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

II. Preparation of Sample

7. **Sample.** The sample as received should be thoroughly stirred and agitated, warming if necessary to insure a complete mixture free from crystallized solids, before the portion for analysis is removed.

8. **Dehydration.** If the presence of 3% water is suspected or known, the oil shall be dehydrated before distillation in accordance with the method for dehydration as given in "Standard Method for the Determination of the Amount of Water in Creosote Oil" (See MANUAL, Creosote, Analysis, Water Content). In all cases of dispute the oil shall be dehydrated before distillation.

III. Procedure

9. **Assembling Apparatus.** The flask shall be supported on a tripod or rings over two sheets of 20-mesh gauze, 150 mm. square, as shown in Fig. 3. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the tubulature with the bottom of the bulb 12 to 13 mm. from the surface of the oil in the retort. The exact location of the thermometer bulb shall be determined by placing a vertical rule graduated in divisions not exceeding 1 mm. back of the retort, when the latter is in position for the test, and sighting the level of the liquid and the point for the bottom of the thermometer bulb.

The axis of the bulb through the tubulature shall be vertical.

The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner should be protected from draughts by a suitable shield or chimney, see Fig. 3.

10. **(c) Distillation Test.** One Hundred grams (100g. \pm 0.1g.) of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in 10 minutes \pm 100 seconds.¹ The rate shall be adjusted to 90 to 100 drops per minute within 2 minutes after the first drop and shall be maintained at 80 to 100 drops per minute throughout the distillation.² The distillate shall be collected in weighed receivers. The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates.

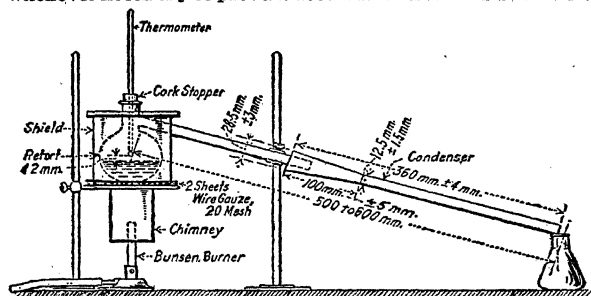


Fig. 3.—Distillation Apparatus Assembly

Fractions shall be collected at the points designated by the specifications. The receivers shall be changed when the thermometer indicates the dividing temperature of each fraction. When the thermometer indicates 355° C., the flame and shield cover shall be removed immediately and any distillate which has condensed in the condenser tube shall be drained into the last fraction.

(b) The residue shall remain in the flask with the cork and the thermometer in position until no vapors are visible; it shall then be weighed. The flask shall then be tipped around so that the residue will flow around the sides, thus collecting any condensed vapors that have collected on the sides of the flask, after which the residue shall be poured into the brass collar used in the float test or into a suitable receptacle and covered. If the residue becomes so cool that it cannot be poured readily from the flask, it shall be reheated to a temperature not exceeding 125° C. by holding the bulb of the flask in a suitable bath and not by the application of flame.

(c) For weighing the receivers and fractions, a balance accurate to at least 0.05 g. shall be used.

(d) During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

(e) The following fractions should be reported:—

0 — 210° C.	270 — 315° C.
210 — 235° C.	315 — 355° C.
235 — 270° C.*	Residue.

If the elevation above sea level at which the distillation is to be made exceeds 1,000 feet, the temperatures at which the fractions are taken should be corrected according to the following table:

TEMPERATURES AT WHICH FRACTIONS SHOULD BE CUT TO CORRECT DISTILLATION TEMPERATURES FOR DIFFERENT ALTITUDES
Corrections are made to the nearest 1° C.

Elevation Above Sea Level in Feet	Fractionation Temperatures for Various Altitudes					
0	200° C.	210° C.	235° C.	270° C.	315° C.	355° C.
1,000	198	208	233	268	313	353
1,500	197	207	232	267	312	352
2,000	196	206	231	266	311	351
2,500	196	206	230	265	310	350
3,000	195	205	230	264	309	349
3,500	194	204	229	263	308	348
4,000	193	203	228	263	307	347
4,500	193	202	227	262	306	346
5,000	192	202	226	261	305	344
5,500	191	201	225	260	304	343
6,000	190	200	225	260	303	343

(f) If the fraction 0—210° C. contains water the amount of water shall be determined. This amount shall then be deducted from the weight of oil taken and all of the fractions shall be corrected to a percentage based on dry oil.

A convenient method for determining the amount of water is to transfer this fraction after weighing to a tube or cylinder graduated in one-tenth c. c.'s and add benzol. This almost always causes a very clean separation between the oil and the water.

In cases of routine or plant control analyses where the presence of water is known or suspected, the oil shall be heated slowly until a temperature of 150° C. is reached. The heat shall then be increased and so regulated that the specified distillation rate will be reached in 5 minutes. From this point the method as specified shall be followed.

Unless care is taken, the rate of distillation will fall below the specified rate during the last fraction. It is necessary to continue the proper rate throughout the distillation in order not to influence the results of tests of residue.

The fraction 235–270° C. is not usually required in creosote specifications, but it is recommended that this cut be made, since it gives useful information and does not materially increase the operator's labor.

Amended, 1933, Revised 1934 (Editorial revisions).

11. CRUDE ANTHRACENE

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least 10 ounces (about 300 g), passing a $\frac{1}{16}$ inch mesh sieve, shall be supplied for carrying out the following and any other tests which may be required by the particular specification which is being followed. The sample shall be supplied and stored in an air-tight non-absorbent container.

SERIAL No.: C.A. 1-38

Anthracene

(After Z. anal. Chem., 1877, 16, 61.)

General:

The Committee has again taken note of the alternative variations of this test which have appeared in the literature but does not consider it desirable to vary from the following procedure.

The method is based on the conversion of the anthracene in the sample to anthraquinone and the isolation of the latter in the pure state.

Applicability:

The test described below is only applicable to samples of crude anthracene containing at least 35 per cent but not more than 85 per cent of anthracene (see Note).

Apparatus:

An anthraquinone flask consisting of a flask of about 500 ml capacity, fitted with an air-cooled reflux condenser 750 mm ± 10 in length and 10 mm ± 1 in internal diameter, the lower end making a ground-in connexion with the neck of the flask and being finished with a drip end (Fig. 27, page 300).

A flat bottomed thin porcelain dish about 90 mm in diameter, completely glazed internally and externally, and weighing not more than 50 g.

Method:

One gram of the sample shall be weighed as rapidly as possible in order to avoid loss of water and other volatile constituents, and transferred to the anthraquinone flask; 45 ml of glacial acetic acid shall be added and the condenser fitted to the neck of the flask. The flask shall be heated by means of a small flame, bringing the liquid to the boiling-point to complete the solution of the anthracene.

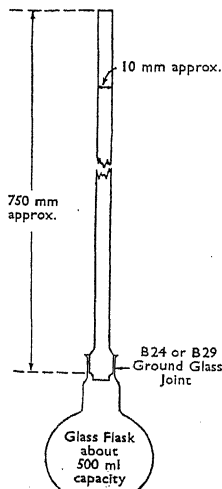


Fig. 27.—Anthracene in Crude Anthracene—Anthraquinone Flask.

The contents of the flask shall be kept boiling at such a rate that the ring of condensate is not more than half way up the condenser; the necessary slow rate of heating can conveniently be maintained by using a micro-burner. While the boiling rate is so maintained, a solution made by dissolving 15 g of pure chromic acid in 10 ml of distilled water and adding 10 ml of glacial acetic acid shall be added gradually through the condenser tube from a dropping funnel or by other suitable means. The addition of the chromic acid solution shall extend with approximate uniformity over two hours and the contents of the flask shall be kept boiling gently for a further two hours (see Note).

The flask and contents shall be allowed to stand for at least twelve hours, after which 400 ml of distilled water shall be added, the contents of the flask thoroughly mixed by swirling, and the whole allowed to stand for two hours.

The contents of the flask shall be filtered through a smooth surfaced filter paper (e.g. Whatman No. 52 or 530) about 185 mm in diameter and washed first with cold distilled water until all traces of acid are removed (this takes from 400 to 600 ml of water); then with 100 ml of boiling 1 per cent *w/w* clear aqueous caustic soda solution; finally with boiling distilled water until all traces of alkali are removed (this also takes from 400 to 600 ml of water). During the final washings the crystals shall be collected into the apex of the cone. The filtration and washing must not be aided by the use of suction,

which has a tendency to draw the anthraquinone into the pores of the paper.

The filter paper shall be spread (care being taken not to tear it) upon a glazed porcelain tile and the anthraquinone transferred to the weighed porcelain dish by means of hot water, using a wash bottle with a moderately fine jet. The dish and its contents shall be dried by heating first on a water-bath until all visible water has been removed and finally in an oven at a temperature of 98° to 100°C for ten minute periods until the weight is constant within one milligram. The dish shall be placed in a desiccator during the cooling intervals and shall be weighed when cold. The appearance of the crude anthraquinone, which consists of needle crystals and amorphous particles, shall be noted.

The crude anthraquinone shall be treated with ten times its weight of oleum containing 10 *per cent w/w* of free sulphur trioxide and the mixture heated for ten minutes on a water or steam bath at 100°C. The solution shall then be allowed to stand for at least twelve hours in an atmosphere saturated with water vapour at the laboratory temperature.

The crystalline mass which separates shall be broken up by means of a small glass rod preferably provided with a closed rubber tip, transferred to a beaker with about 200 ml of cold distilled water and after agitation, the whole shall be allowed to stand for two hours. The contents of the beaker shall be filtered through a smooth surfaced filter paper (e.g. Whatman No. 52 or 530) about 185 mm in diameter and washed first with cold distilled water until all traces of acid are removed (this takes from 400 to 600 ml of water); then with 100 ml of boiling 1 *per cent w/w clear* aqueous caustic soda solution; finally with boiling distilled water until all traces of alkali are removed (this also takes from 400 to 600 ml of water).

The refined anthraquinone shall be transferred to the dish previously used, in the manner already described. It shall be dried first on the water-bath and subsequently in an oven at a temperature of 98° to 100°C for ten minute periods, with intermediate cooling in a desiccator and weighing, precisely as specified for the crude anthraquinone. Finally, the dish shall be heated cautiously over a low flame at a temperature just sufficient to volatilize the anthraquinone, avoiding ignition. The loss by volatilization represents the weight (W g) of pure anthraquinone, whence the percentage by weight of anthracene in the sample is 85.6W.

STANDARD METHODS

Note:

Samples containing less than 35 *per cent* of anthracene such as those obtained in the analysis of crude tar (Serial No. C.T. 7, page 77) require for oxidation more chromic acid than that specified under "Method." The addition of chromic acid solution of the prescribed strength shall in such cases be continued until the green colour of the contents of the flask acquires a brown tint.

SERIAL No.: C.A. 2-38

Solid Paraffins

General:

The aromatic constituents of the crude anthracene are removed by heating with a large excess of pure sulphuric acid. The paraffins, which are unattacked, are separated and weighed.

Method:

Twenty grams of a representative portion of the sample shall be weighed into a 500 ml beaker; 290 ml of pure 98 *per cent w/w* sulphuric acid shall be added; the contents of the beaker shall be well mixed and the mixture heated fairly rapidly to 125° to 127°C. The mixture shall be maintained at this temperature for thirty minutes with frequent stirring to ensure effective disintegration and subsequent solution of all aggregates of the material.

The mixture shall be allowed to cool and shall be added cautiously with continuous stirring to about 500 ml of cold distilled water in a large separating funnel (see Note); the beaker shall be washed with 100 ml of boiling distilled water and the washings added to the main bulk, which shall be well mixed and allowed to stand overnight. Accelerated cooling at this stage is not advisable, as paraffins tend to crystallize on the sides of the funnel and in consequence are subsequently less easily washed out with petroleum ether.

Thirty millilitres of redistilled petroleum ether (boiling-point 60° to 80°C) shall be used to rinse the 500 ml beaker and shall then be added to the solution in the separating funnel, which shall be shaken thoroughly and the contents allowed to separate. The separating funnel shall be allowed to stand until *all* froth has disappeared and the acid layer shall be run off and reserved for re-extraction. The petroleum ether layer

shall be run into a 300 ml separating funnel, the sides of the large funnel washed with about 50 ml of distilled water and this washing added to the solution in the smaller separating funnel (see Note).

The main bulk of the acid solution shall be returned to the large funnel and extracted with a further 30 ml of the petroleum ether; the funnel shall be allowed to stand until the froth has disappeared and the contents have separated, and the acid layer shall be run off and reserved for a third extraction. The second petroleum ether extract shall be added to the first as also shall a further 50 ml of distilled water after it has been used to wash the large funnel.

The main bulk of the acid solution shall be again returned to the large funnel and extracted with a third portion of 30 ml of the petroleum ether; the separation of the petroleum ether extract, the washing of the large funnel and the addition of the extract and washings to the smaller funnel shall be carried out as before. The smaller funnel will then contain about 90 ml of petroleum ether extract and about 150 ml of aqueous acid washings. The contents of this funnel shall be swirled gently and allowed to stand for ten minutes, when the acid layer shall be run off. The clear ethereal solution shall be washed as follows, allowing ten to fifteen minutes for separation after each washing:—

- (a) with 30 ml of 5 per cent *w/w* clear aqueous caustic soda solution;
- (b) with four successive portions, each of 8 ml, of concentrated sulphuric acid; and finally,
- (c) with distilled water until free from acid (usually about six washings of 50 to 100 ml each are required).

After the final water wash and separation of water, the petroleum ether solution shall be poured *through the top of the funnel* into a weighed 100 ml beaker or small wide mouthed flask, evaporated on a steam bath until free from the odour of petroleum ether and dried to constant weight at 100°C. If *W* be the weight in grams of solid paraffins obtained, the percentage by weight of solid paraffins in the sample is 5*W*.

Note:

It is essential that the separating funnels be scrupulously clean and free from grease at the commencement of the test.

12. PITCH

Nature and Quantity of Sample:

The sample shall be prepared in accordance with the section on "Sampling," pages 13 to 30.

At least 2 lb (about 1,000 g) shall be supplied for the full series of tests described in this section; the sample shall be stored in an air-tight container.

SERIAL No.: P.T. 1-38

Specific Gravity; Density

Method:

The specific gravity or density may be determined by any recognized method (see Serial No. G.P. 2, Part II, page 61 (specific gravity of solids) or Serial No. G.P. 1, Part II, page 46 (density of solids)).

Softening or Melting of Pitch

General:

The softening and melting of pitch do not take place at definite temperatures; as the temperature rises, the material gradually changes from a brittle or viscous and slowly flowing material to a softer and less viscous substance. For this reason, determinations relating to the softening or melting of pitch must be made by empirical but closely defined methods, which must be strictly adhered to if the results from any one particular method are to be comparable.

While they do not constitute determinations of precisely the same characteristic or group of characteristics, it remains the fact that there are in continuing demand four methods of test relating to this group of characteristics; each uses a different set of conditions and measures a different stage in the gradual process of softening or melting. The standard technique for each of these methods is described below in the order:—

Twisting Test	Serial No. P.T. 2
Kraemer and Sarnow Test	„ P.T. 3
Ring and Ball Test	„ P.T. 4
Half Inch Cube Test	„ P.T. 5

Twisting Test

(After H. Frankland Taylor, *J. Soc. chem. Ind.*, 1926, 45, 423t.)

Apparatus:

A Frankland Taylor pitch testing unit as shewn in Figs 28 and 29, below and opposite.

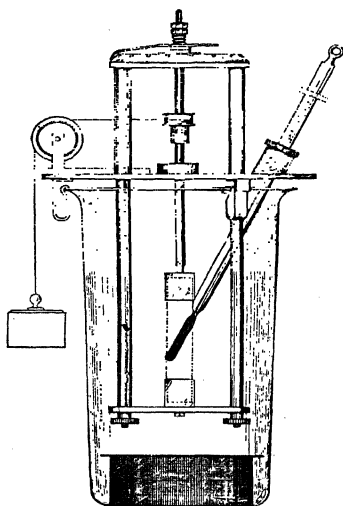


Fig. 28.—Twisting Test for Pitch—
Assembled Apparatus.

The apparatus is constructed in brass and consists of a device for the application of a standard torque to a square prism of pitch which is being heated at a uniform rate. The torque is exerted by a weight of 150 g suspended by a thread which, after passing over a pulley, is attached to a drum, the circumference of which is equal in dimension to the perimeter of a cross section of the pitch prism, viz. 51 mm. The drum is fixed to a vertical spindle which is free to rotate. A square socket, 13 mm \times 13 mm \times 15 mm deep, is fitted to the lower end of the spindle and the upper end of the prism of pitch is fitted into the socket; the lower end rests in a similar socket which is stationary, being attached to the bottom horizontal member of the framework.

The distance between the two sockets is exactly 50 mm. The spindle can be raised to permit insertion of the pitch sample. A disc, graduated in degrees of arc, is fixed to the upper horizontal member of the framework and a pointer is fitted at the top of the vertical spindle so that the angle of rotation can be measured. The pointer is so arranged that it can be adjusted to indicate zero at the beginning of each experiment. A small collar supports the vertical spindle on the upper horizontal section of the framework. Ball bearings are provided to ensure free movement; they must be well lubricated (using a *very thin* oil) and maintained in good condition.

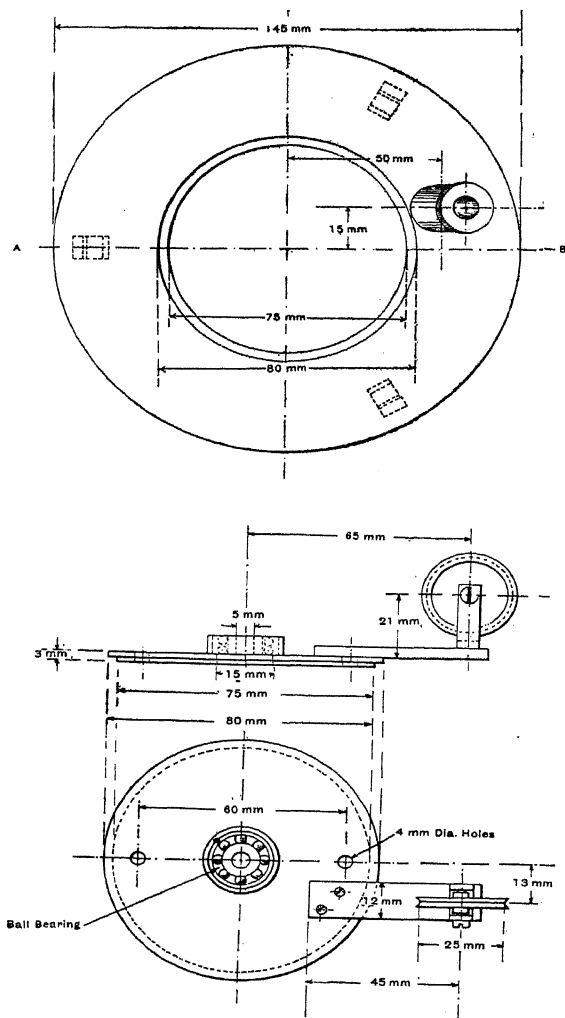
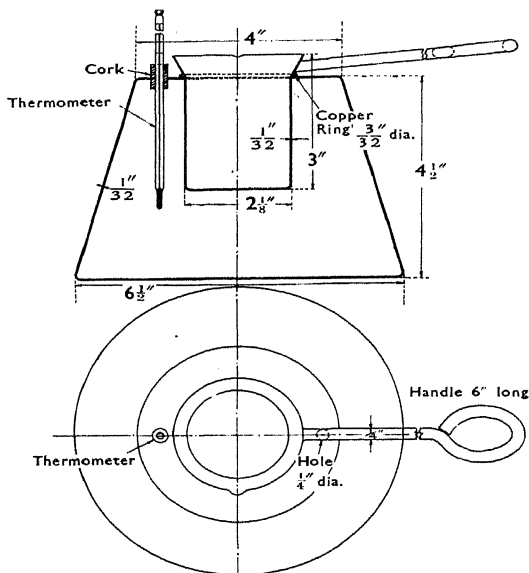


Fig. 29.—Twisting Test for Pitch—
Supporting Plate, etc.,

A standard thermometer having the range 0° to 120°C (Schedule No. T3a, page 333).

A wire gauze disc supported horizontally on a wire gauze cylinder, 25 mm in height. This serves to break up convection currents.



Dimensions are intended for guidance only.

Fig. 30.—Pitch Melting Bath—Elevation and Plan.

A heating bath consisting of a 1,500 ml beaker, of tall form without spout.

A Bunsen burner.

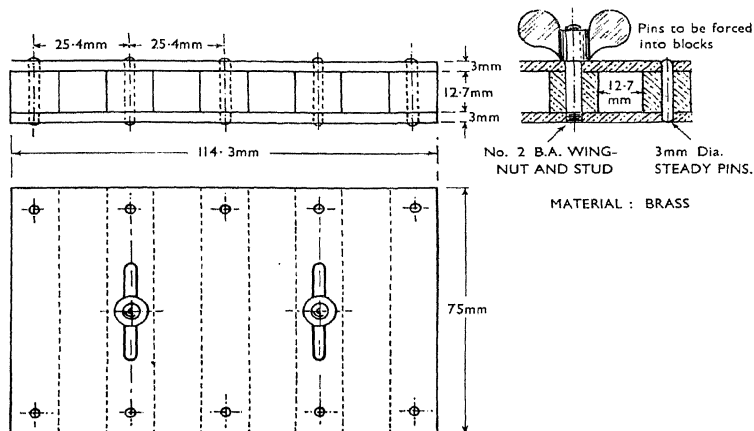
A pitch melting bath (Fig. 30, above) consisting of an oil bath made of about 22 gauge copper sheet, supporting a smaller copper vessel in which the pitch is melted; the smaller vessel is fitted with a brass handle.

A brass pitch mould and plate; a suitable form of mould is shewn with the necessary dimensions in Fig. 31, opposite; as indicated in Note i to Serial No. P.T. 5, page 322, the mould shewn in Fig. 31 is designed to serve for the half inch cube

and the present tests; a mould for making only two prisms would serve equally well for the present test.

Method:

A portion of the sample, weighing at least 60 g and powdered if practicable, shall be placed in the inner vessel of the pitch melting bath, the outer vessel of which contains oil at about 80 Centigrade degrees above the expected result of the test. The pitch shall be stirred gently during the heating period



The dimensions are approximate, except those shewn as 12.7 mm which must be adhered to within ± 0.05 mm.

Fig. 31.—Twisting Test for Pitch—Pitch Mould.

to ensure homogeneity and the elimination of air bubbles. As soon as the temperature of the pitch is between 65 and 70 Centigrade degrees above the expected result of the test, the prisms of pitch shall be formed as described below.

The inner surfaces of two of the cavities of the mould and one side of the brass plate shall be amalgamated, the mould assembled and placed with one end resting on the amalgamated side of the brass plate, and the pitch shall be poured into the amalgamated cavities in a thin stream. The quantity of pitch used shall be such that, after ten minutes, a slight excess of pitch remains above the level of the mould. After cooling for ten minutes, it is permissible to hasten the subsequent cooling by immersion of the mould under running water.

When the mould has cooled to laboratory temperature, the excess of pitch shall be removed by means of a knife and without the application of heat.

If for any reason the test has to be repeated, the pitch melting bath shall be cleaned, a fresh portion of the sample heated and further prisms made as described above.

One of the prisms (75 mm \times 12.7 mm \times 12.7 mm) shall be fitted into the two brass sockets of the Frankland Taylor machine. It is permissible to facilitate the subsequent removal of the prism from the sockets by rubbing chalk over the surfaces of the pitch prism which will come into contact with the sockets, i.e. over the ends of the pitch prism and along its length for a distance of 15 mm from each end.

About 1,450 ml of water at about 20°C shall be measured into the beaker and the gauze disc and cylinder shall be introduced. The pitch testing unit shall be placed in position as shewn in Fig. 28, page 306; the level of the water shall be adjusted if necessary by the addition of more water, e.g. from a pipette, so that it is at least 25 mm above the junction of the vertical spindle and the upper of the two sockets carrying the pitch prism. The thermometer shall be fitted as shewn in Fig. 28.

The apparatus shall be allowed to stand for fifteen minutes in order that the pitch may attain the temperature of the surrounding water. Heat shall then be applied in such a manner that the temperature is raised two Centigrade degrees in each minute; this rate shall be maintained within the limits 30 seconds \pm 5 for each Centigrade degree rise after at least 20 Centigrade degrees below the expected result and shall not merely be the average over the period of the test. Rigid adherence to the specified rate is essential and all tests in which the rate in any half minute period is outside the limits shall be rejected.

When the temperature commences to rise, the torsion, which shall not have been applied prior to this stage, shall be imposed by allowing the weight to hang freely and vertically. The pointer shall immediately be set to the zero mark on the scale. The temperature at which the pointer indicates 180° rotation shall be reported.

It is advisable to carry out duplicate tests and in consequence provision is made for the preparation of a second pitch prism. If duplicate readings for the same sample differ, they shall each be reported as well as the mean.

Kraemer and Sarnow Test (S.T.P.T.C. Modification)

Apparatus:

The apparatus shall be as shewn in Figs 32 to 34, pages 311 to 314, and as described below:—

The water-bath consists of a 600 ml beaker and a 250 ml beaker, both of tall form without spout. The beakers are

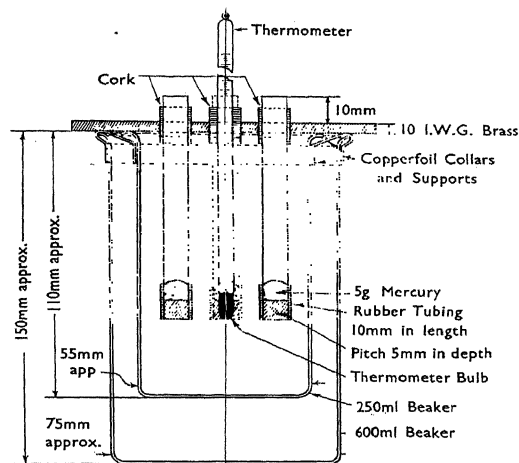


Fig. 32.—Kraemer and Sarnow Test for Pitch—
Assembled Apparatus.

mounted one within the other and with their upper edges in the same plane; this may conveniently be effected by making a support as follows:—

A strip of copper foil, about 12 mm wide, is placed round the outside of the smaller beaker and is soldered at the overlap to form a collar. The widest beaker available of this form and capacity (250 ml) is selected for the purpose, so that it can be replaced readily in the event of breakage. Three other strips of copper foil, also about 12 mm wide, are soldered to the collar, at right angles to its length and at equal distances apart. A similar collar is prepared for the larger beaker and,

with the smaller one turned upside down and the larger one placed concentrically over it, the three strips from the smaller collar are soldered, when pulled tight, to the larger collar.

The mercury tube support consists of a disc of 10 I.W.G. brass plate, about 100 mm in diameter, bored with five holes, each 12.5 mm in diameter and so placed that one is in the exact centre and the others about equidistant from it and also from one another. Their distance from the centre of the disc is determined by the internal diameter of the inner beaker, their positions being such that the mercury containers which are to pass through them shall be situated as nearly as possible midway between the centre and wall of the beaker. It is advisable to number the radial holes 1, 2, 3 and 4 for identification purposes during the test.

The mercury containers consist of glass tubes, 95 mm in length and 6.5 mm ± 0.1 in internal diameter. They shall be about 8 mm in external diameter and one end of each must be accurately ground at right angles to the axis.

The pitch containers are short lengths of brass or stainless steel tubing, 6.5 mm ± 0.1 in internal diameter. They shall be ground level at their ends, at right angles to their length, which must be 5.0 mm ± 0.2 . The tubes shall be about 8 mm in external diameter.

A standard thermometer having the range 0° to 120°C (Schedule No. T3a, page 333).

A filter paper, cut to the required size and held down by a thin flat metal ring about 6 mm wide and of such diameter as will allow it just to fit the inner beaker, may be employed to catch the molten pitch and prevent it from contaminating the bottom of the beaker.

The apparatus is assembled on a tripod and is protected by a standard draught screen (Schedule No. S1, page 372) of sufficient height for the purpose when standing on the bench.

A Bunsen burner, with a governor if the gas supply is liable to fluctuate.

A pitch melting bath (Fig. 30, page 308) identical with that described in Serial No. P.T. 2, page 306.

A mercury pipette (Fig. 33, opposite) consisting of a three-way, oblique bore, accurately ground stopcock, having tubes about 1.5 mm in internal diameter and about 6 mm in external diameter. The delivery tube is tapered to allow it to enter

the glass mercury containers freely. The tube adjacent to the delivery tube is bent at right angles and is connected, by means of rubber pressure tubing, with a small open mercury levelling reservoir. A small bulb is blown on the tube at the opposite side of the stopcock and at about 25 mm therefrom. A length of enamel backed thermometer capillary tubing about 0.6 mm in internal diameter is fused on the upper end of the bulb.

The calibration of the pipette is carried out as follows, if possible when the temperature of the laboratory is about 15°C:—

The glass parts must be thoroughly cleaned by successive treatments with chromic acid, caustic soda, alcohol and dry ether. No lubricant may be applied to the stopcock as this inevitably works its way into the bore holes. Mercury from the reservoir is introduced slowly into the small bulb and up the capillary stem. The stopcock is closed against the reservoir and the delivery tube, and the level of the mercury in the capillary tube is marked by means of Indian ink, using a finely pointed pen. The stopcock is opened to the delivery tube and the mercury which flows out is received in a small accurately weighed vessel. A rubber bulb is connected to the upper end of the capillary tube and the small particles of mercury still adhering to the walls are blown into the weighed vessel. The vessel and mercury are weighed and the process repeated until a point is found on the capillary tube, from which $5.00 \text{ g} \pm 0.05$ of mercury are discharged in each of six successive experiments.

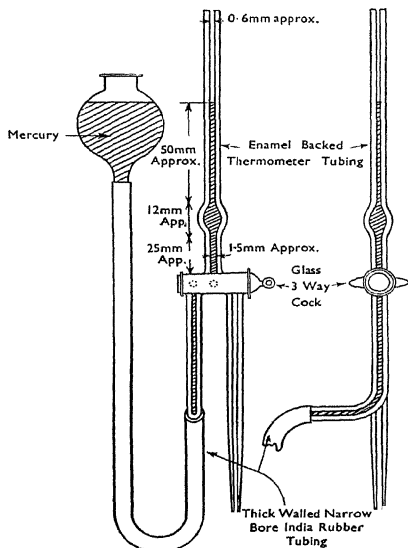
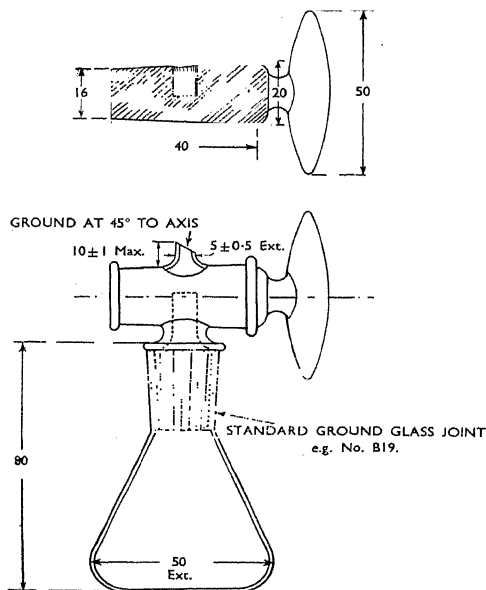


Fig. 33.—Kraemer and Sarnow Test for Pitch—Mercury Pipette.

The mercury measure shewn in Fig. 34, page 314, may be used in place of the mercury pipette described above. The

mode of operation of the measure is obvious from the illustration. The cavity in the plug of the stopcock shewn in Fig. 34 shall be of such dimensions (about 7 mm in diameter and about 9.5 mm in depth) that the unit will deliver $5.00 \text{ g} \pm 0.05$ of mercury at 20°C .



All dimensions in millimetres and, where not provided with tolerances, intended for guidance.

Fig. 34.—Kraemer and Sarnow Test for Pitch—
Alternative Mercury Measure.

Method:

A portion of the sample, weighing at least 40 g and powdered if practicable, shall be placed in the inner vessel of the pitch melting bath, the outer vessel of which contains oil at about 60 Centigrade degrees above the expected result of the test. The pitch shall be stirred gently during the heating period to ensure homogeneity and the elimination of air bubbles. As soon as the temperature of the pitch is between 45 and 50

Centigrade degrees above the expected result of the test, the pitch containers shall be filled as described below.

The pitch containers shall be placed on an amalgamated brass plate and the pitch poured into them in a thin stream and allowed to overflow so that the containers are encased in pitch. After cooling for ten minutes, the excess of pitch shall be broken away, that adhering to the sides being scraped off and that in excess over the end trimmed to the level of the brass tube by means of a knife and without the application of heat.

If for any reason the test has to be repeated, the pitch melting bath shall be cleaned, a fresh portion of the sample heated and the pitch containers filled as described above.

The pitch containers shall be attached to the mercury containers by means of 10 mm lengths of rubber tubing. The ground end of each glass tube must be brought into close contact with that end of the pitch container which was uppermost while being filled and the rubber connexions must reach exactly to the lower end of the pitch containers. The glass tubes shall be fitted at their other ends into corks to support them in the holes of the brass plate and each of the tubes shall be charged with 5 g of mercury. They shall be fixed into the holes marked 1, 2, 3 and 4 in the brass support in such a way that they are held vertically and that 10 mm of their length projects above the upper surface of the plate. The thermometer shall be fixed vertically in the central hole so that the bottom of the bulb is level with the lower surface of the pitch and central to the four tubes.

The support shall be placed in the water-bath, into the larger and smaller beakers of which have been placed 360 ml and 200 ml respectively of freshly boiled distilled water which has been cooled to at least 30 Centigrade degrees below the expected result (see Note i). The support shall be so placed on the inner beaker that the thermometer lies along the axis.

The tubes shall be allowed to stand for fifteen minutes in order that the pitch may attain the temperature of the surrounding water. Heat shall then be applied in such a manner that the temperature is raised two Centigrade degrees in each minute; this rate shall be maintained within the limits 30 seconds ± 1 for each Centigrade degree rise after at least 20 Centigrade degrees below the expected result, and shall not

merely be the average over the period of the test. Rigid adherence to the specified rate is essential and all tests in which the rate in any half minute period is outside the limits shall be rejected.

The temperature at which the mercury bursts through the envelope of viscous pitch shall be reported. Should the envelope of pitch reach the bottom of the beaker without bursting, the temperature at which it reaches midway between the glass tubes and the bottom of the beaker shall be reported. If the readings differ as between portions of the same sample in the several containers, they shall each be reported as well as the mean.

In cases where it is possible to allow samples to accumulate until four can be tested together, the following practice should be adopted:—

Four tubes from each sample are prepared, and these are tested in batches of four, consisting of one tube from each sample, maintaining a steady increase of temperature, and noting accurately the average time per one degree rise in temperature. If possible, it should be arranged that two sets take slightly more than thirty seconds per degree rise, and two slightly less, each rate of heating lying, of course, within the permissible tolerance. The readings obtained for each sample are plotted against the time in seconds for each rise of one degree; a curve is drawn and the point at which this curve crosses the thirty seconds line is taken as the correct result.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 0.5 Centigrade degree of the average of the several readings when the readings do not exceed 100°C and within ± 1.0 Centigrade degree of the average when the readings exceed 100°C but do not exceed 120°C .

Notes:

(i) In the case of hard pitches which by the above procedure would shew a result above 80°C , the water in the beakers shall be replaced by neutral glycerine and the pitch containers shall be warmed before pouring in the pitch.

(ii) It must not be assumed that the foregoing method gives the same results as any of the standard Continental methods of carrying out the Kraemer and Sarnow test.

Ring and Ball Test

Apparatus:

The apparatus shall be as shewn in Fig. 35, page 318, and as described below:—

The tapered pitch rings (Fig. 36, page 318) are made in brass to the following dimensions:—

	<i>mm</i>	<i>inch</i>
Depth	6.35 \pm 0.10	1/4
Internal diameter at bottom	15.88 \pm 0.10	5/8
Internal diameter at top ..	17.46 \pm 0.10	11/16
External diameter above shoulder	20.60 \pm 0.10	13/16
External diameter below shoulder	18.45 \pm 0.45	23/32

The rings are provided with a support, a convenient form being that shewn in Figs 35 and 37; the support consists essentially of two brass plates, the upper one in the form and to the dimensions shewn in Fig. 37, and designed to carry two rings. The depth of the pitch rings below the shoulder is the same as the thickness of the ring supporting plate. The lower plate is about 25 mm in width and about 75 mm in length and is fixed so that its upper surface is 25 mm \pm 0.5 below the lower surface of the ring supporting plate. The balls which rest on the pitch in the rings are of steel and have a diameter of 9.53 mm and a weight of 3.50 g \pm 0.05 each. The thermometer has the range 0° to 120°C and is graduated for total immersion (Schedule No. T3b, page 333).

The heating bath consists of an 800 ml beaker, of squat form without spout.

The assembled apparatus is supported on a tripod as shewn in Fig. 35, page 318. The tripod carries a flame screen of wire gauze (about 20 mesh per inch) and is covered with a square of wire gauze (about 20 mesh per inch). Heating is effected by means of a Bunsen flame. The effect of draughts shall be eliminated by using a suitable draught screen if necessary.

A pitch melting bath (Fig. 30, page 308) identical with that described in Serial No. P.T. 2, page 306.

A plate for moulding, of the dimensions shewn in Fig. 38, page 319.

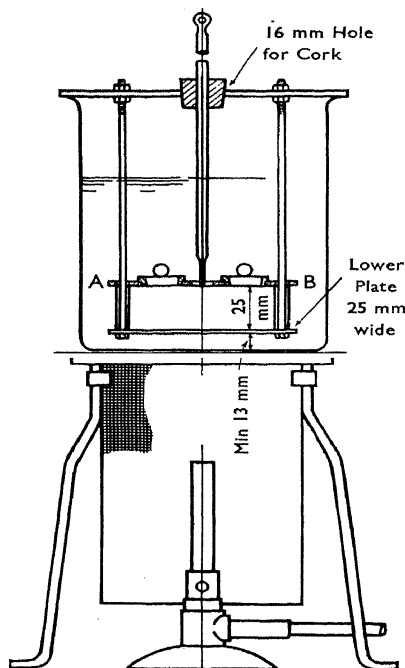


Fig. 35.—Ring and Ball Test for Pitch—Assembled Apparatus.

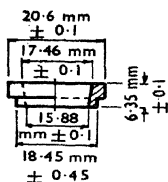


Fig. 36.—Ring and Ball Test for Pitch—Pitch Ring.

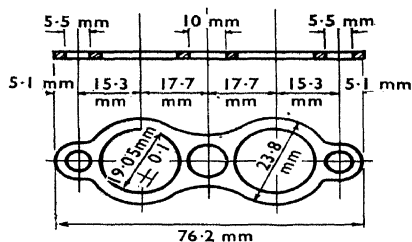


Fig. 37.—Ring and Ball Test for Pitch—Ring Support.

Method:

The pitch rings shall be placed in an oven at a temperature between 95° and 110°C, where they shall remain until required for filling.

A portion of the sample, weighing at least 40 g and powdered if practicable, shall be placed in the inner vessel of the pitch melting bath, the outer vessel of which contains oil at about 50 Centigrade degrees above the expected result of the test. The pitch shall be stirred gently during the heating period to ensure homogeneity and the elimination of air bubbles. As soon as the temperature of the pitch is 35 to 40 Centigrade degrees above the expected result of the test, the rings shall be filled as described below.

One of the warmed pitch rings shall be placed inverted within each of the sets of guide pins on the moulding plate (which may, if desired, have been amalgamated to prevent adhesion of the pitch) and the pitch poured into the rings in a thin stream. The quantity of pitch used for each ring shall be such that, after twenty minutes, a slight excess of pitch remains above the level of the pitch ring. At the end of the cooling period this excess shall be removed by means of a knife and without the application of heat.

If for any reason the test has to be repeated, the pitch melting bath shall be cleaned, a fresh portion of the sample heated and the rings filled as described above.

Six hundred millilitres of freshly boiled and cooled distilled water shall be placed in the beaker (see Note i). The filled rings shall be fitted into the support and the latter placed concentrically in the beaker. The thermometer shall be inserted and so adjusted that it lies along the axis of the beaker; the bottom of the thermometer bulb shall be level with the bottom of the rings and the bulb shall be central with the central hole of the ring supporting plate. The balls shall be placed in the beaker but not on the pitch in the rings. The level of the water in the beaker should then be at least

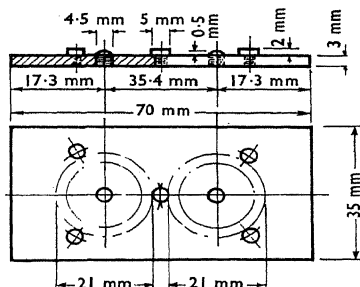


Fig. 38.—Ring and Ball Test for Pitch—Moulding Plate.

51 mm above the top of the pitch rings; if this be not so, the beaker shall be replaced by one which, with 600 ml of water in it and the pitch ring support and other fittings in position, meets this requirement.

The apparatus shall be set so that the ring supporting plate is horizontal. The apparatus shall be brought to and maintained for fifteen minutes at a temperature at least 45 Centigrade degrees below the expected result, except that, if the expected result be lower than 50°C, the temperature of the apparatus shall be adjusted to and maintained for fifteen minutes at 5°C. The balls shall be placed, by means of tongs, in the central indentations on the upper surface of the pitch in the rings.

The burner shall be placed under and midway between the centre and the edge of the beaker and on a diameter at right angles to that which includes the rings and the thermometer. Heat shall be applied in such a manner that the temperature is raised five Centigrade degrees each minute; this rate shall be maintained within ± 0.5 Centigrade degree over each one minute period after the first three, and shall not merely be the average over the period of the test. Rigid adherence to the specified rate is essential and all tests in which the rate in any one minute period is outside the limits shall be rejected.

The temperature at which the pitch surrounding the steel ball first touches the lower plate of the support shall be reported. If duplicate readings for the same sample differ in the two rings, they shall each be reported as well as the mean. No correction shall be made for the emergent stem of the thermometer (see Note ii).

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 0.5 Centigrade degree of the average of the several readings when the readings do not exceed 100°C, and within ± 1.0 Centigrade degree of the average when the readings exceed 100°C but do not exceed 120°C.

Notes:

(i) When testing hard pitches which by the above procedure would shew a result above 80°C, the water in the beaker shall be replaced by neutral glycerine.

(ii) The above method is identical with the method using two rings in A.S.T.M. Designation E28-36T, of the American

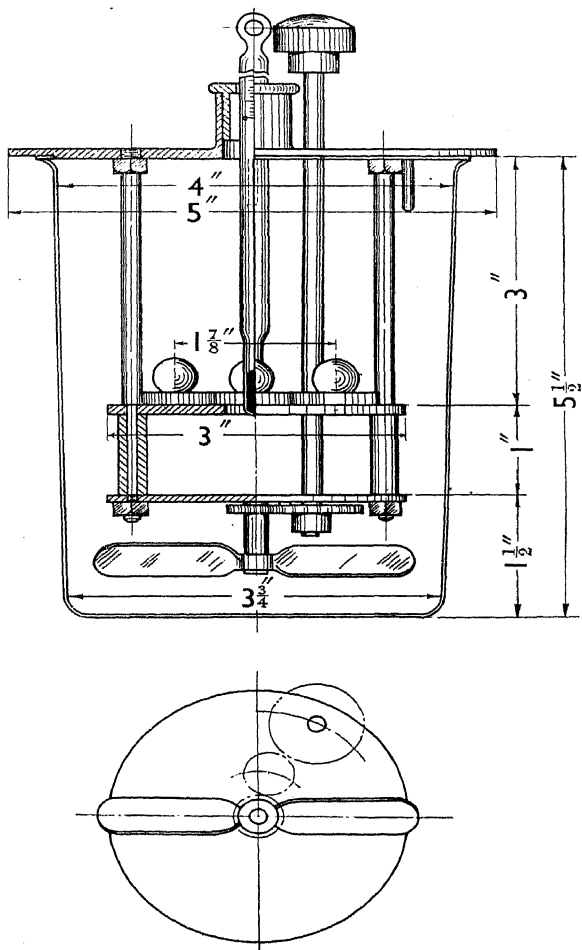


Fig. 39.—Ring and Ball Test for Pitch—Assembled 6-ring Apparatus and Arrangement of Stirring Gears.

Society for Testing Materials, apart from (a) the use of a moulding plate which forms indentations in the pitch whereby the use of a ball centring guide is obviated; (b) the use of a thermometer T3b in general conformity with the Committee's standard thermometer T3a, but graduated for total immersion (a certified thermometer T3b will give the same readings as the A.S.T.M. thermometer when used in the circumstances of the test); and (c) editorial unification with the present publication.

(iii) In laboratories where many samples are tested, it may be found advantageous to use a circular ring support carrying six pitch rings. This support, when used for samples testing over 80°C, should carry a geared stirrer operated by a knob above the upper plate. When using such an apparatus, the burner is placed under the centre of the beaker and not eccentrically. The rings are not at the same distance from the centre as in the standard ring support, but extended tests with such an instrument of the form shewn in Fig. 39, page 321, have shewn that it gives results in sufficiently practical agreement with those using the standard two-ring instrument to warrant its use for routine operations.

SERIAL No.: P.T. 5-38

Half Inch Cube Test

Applicability:

This method is not recommended for use with samples having a specific gravity below 1.2.

Apparatus:

The apparatus shall be as shewn in Figs 40 to 42, pages 323 to 325, and as described below:—

An enamelled iron bowl as shewn in Fig. 40, opposite, supported on a tripod and filled to the depth of 100 mm with freshly boiled water cooled to 25°C.

A Bunsen burner fitted with a rose.

A cylindrical screen of wire gauze, standing inside the feet of the tripod and reaching above the top of the Bunsen flame.

A ring from which the cubes of pitch are suspended, and having the dimensions shewn in Fig. 41, page 324.

A standard thermometer having the range 0° to 120°C (Schedule No. T3a, page 333), fixed so that it passes through the centre of the ring supporting the pitch and so that the

bottom of the bulb is level with the lower horizontal faces of the cubes.

Copper wire, No. 18 I.W.G.

A pitch melting bath (Fig. 30, page 308) identical with that described in connexion with method Serial No. P.T. 2, page 306.

A brass pitch mould and plate; a suitable form of mould is shewn with the necessary dimensions in Fig. 42, page 325 (but see Note i).

Method:

A portion of the sample, weighing at least 60 g and powdered if practicable, shall be placed in the inner vessel of the pitch melting bath, the outer vessel of which contains oil at about 50 Centigrade degrees above the expected result of the test. The pitch shall be stirred gently during the heating period to ensure homogeneity and the elimination of air bubbles. As soon as the temperature of the pitch is between 35 and 40 Centigrade degrees above the expected result of the test, the cubes shall be formed as described below.

The inner surfaces of the cavities of the mould and one side of the brass plate shall be amalgamated, the mould assembled and placed on the amalgamated side of the brass plate, and the pitch poured into the amalgamated cavities in a thin stream. The quantity of pitch used shall be such

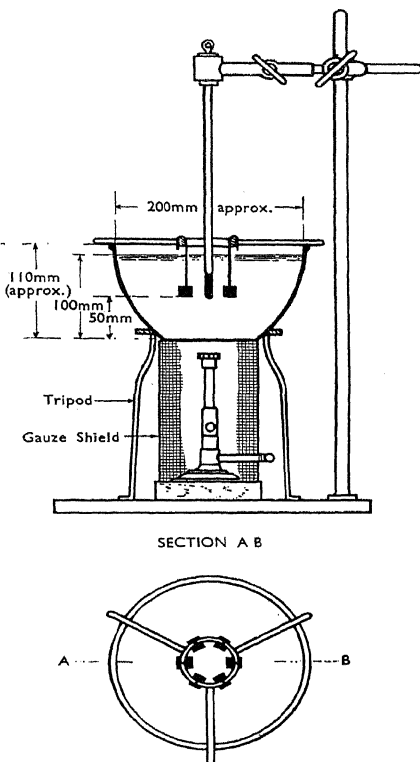


Fig. 40.—Half-Inch Cube Test for Pitch—Elevation and Plan of Assembled Apparatus.

that after ten minutes a slight excess of pitch remains above the level of the mould. After cooling for ten minutes, it is permissible to hasten the subsequent cooling by immersion of the mould under running water. When the mould has cooled to laboratory temperature, the excess of pitch shall be removed by means of a knife and without the application of heat (see Note i).

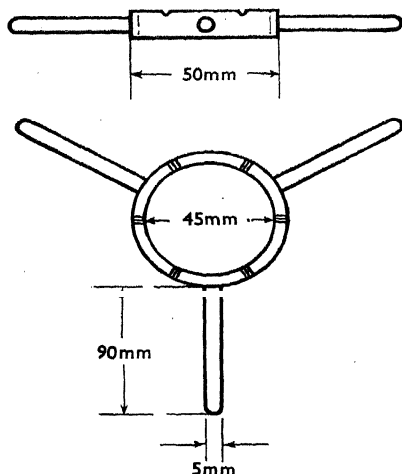


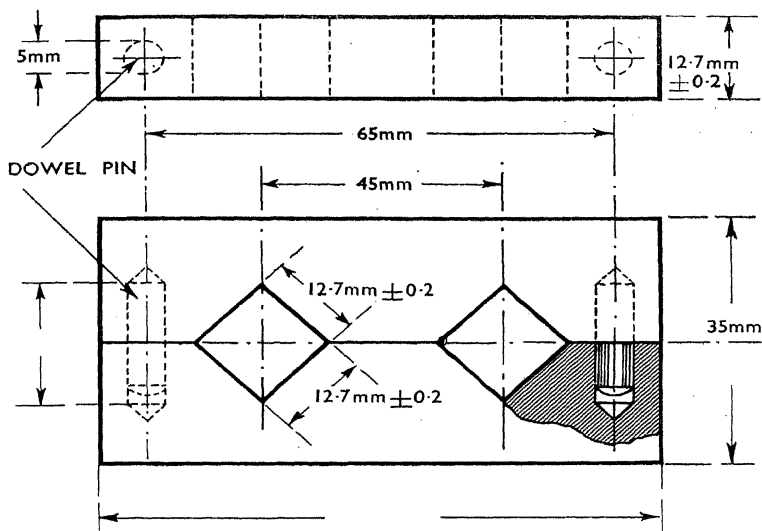
Fig. 41.—Half Inch Cube Test for Pitch—
Brass Ring Support.

If for any reason the test has to be repeated, the pitch melting bath shall be cleaned, a fresh portion of the sample heated and further cubes made as described above.

A piece of the copper wire, about 80 mm in length, shall be bent over at one end to form a hook by which it is subsequently suspended (see Fig. 40, page 323); the longer arm of the hook shall be adjusted in length so that when so suspended the lower end is 50 mm above the bottom of the bowl. The wire shall be heated by placing it in a glass or metal tube, about 2 mm in internal diameter and closed at one end, the tube being immersed in a bath containing heavy lubricating oil at 200° to 250°C. The hot wire shall be withdrawn and passed through the centres of two opposite faces of one of the cubes,

the end being adjusted precisely flush with the lower face. The cube shall be suspended in the water at 25°C, with the lower face horizontal and 50 mm above the bottom of the bowl. The other five cubes shall be similarly mounted on lengths of the copper wire and suspended in the water.

The apparatus shall be allowed to stand for fifteen minutes in order that the pitch may attain the temperature of the surrounding water. Heat shall then be applied in such a



The dimensions other than 12.7 mm ± 0.2 are for guidance.

Fig. 42.—Half-inch Cube Test for Pitch—Pitch Mould.

manner that the temperature is raised two Centigrade degrees in each minute; this rate shall be maintained within the limits 30 seconds ± 5 for each Centigrade degree rise after at least twenty Centigrade degrees below the expected result and shall not merely be the average over the period of the test. Rigid adherence to the specified rate is essential and all tests in which the rate in any half minute period is outside the limits shall be rejected.

The temperature at which the pitch touches the bottom of the bowl shall be reported (see Note ii). If the readings differ

as between portions of the same sample in the several cubes, they shall each be reported as well as the mean. Any cubes dropping in an irregular manner shall not be included in the average, but shall be noted and reported separately.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 0.5 Centigrade degree of the average of the several readings.

Notes:

(i) The pitch mould shewn in Fig. 31, page 309, is for convenience designed to provide in all two prisms of pitch for the twisting test (Serial No. P.T. 2, page 306) and two prisms from each of which three half-inch cubes may be prepared for the present test. If such prisms are used for preparing the cubes, three pieces, each rather more than half an inch in length, shall be broken off each prism. The broken faces shall be rubbed on a flat file flooded with water, until each piece of pitch is $12.7 \text{ mm} \pm 0.2$ in length when measured by gauge.

(ii) The upper limit to which this test may be regarded as accurate lies between 85° and 90°C .

SERIAL No.: P.T. 6-38

Volatile Matter

Apparatus:

The apparatus is shewn in Fig. 43, opposite, and described below:—

A platinum crucible complying with the following requirements:—

External diameter at base	24.0 mm	± 0.5
External diameter at top	34.0 mm	± 0.25
External height	35.0 mm	± 0.5
Volume to brim	27.0 ml	± 0.5
Weight	22.0 g	± 1.0

The crucible shall be provided with a closely fitting lid, grooved to receive the rim of the crucible and having a central hole 2 mm in diameter, as shewn in Fig. 44, page 327.

A Meker burner having a diameter of 31 mm at the top; the flame diameter is 30 mm.

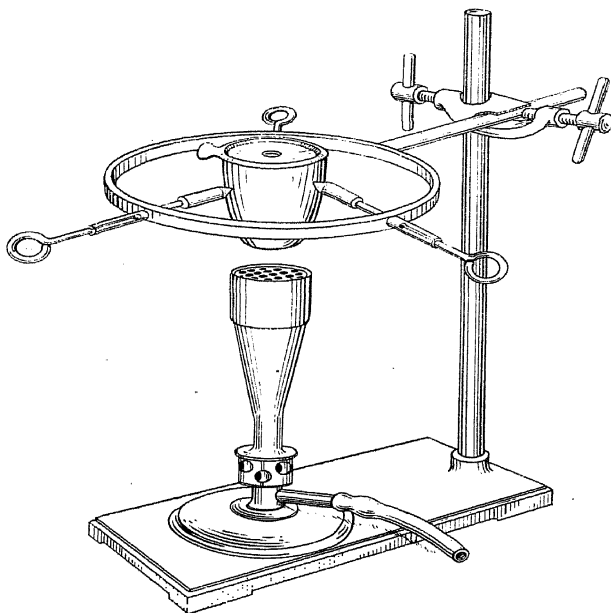


Fig. 43.—Volatile Matter in Pitch—Assembled Apparatus.

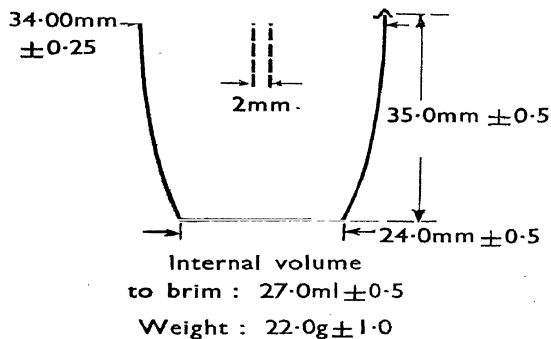
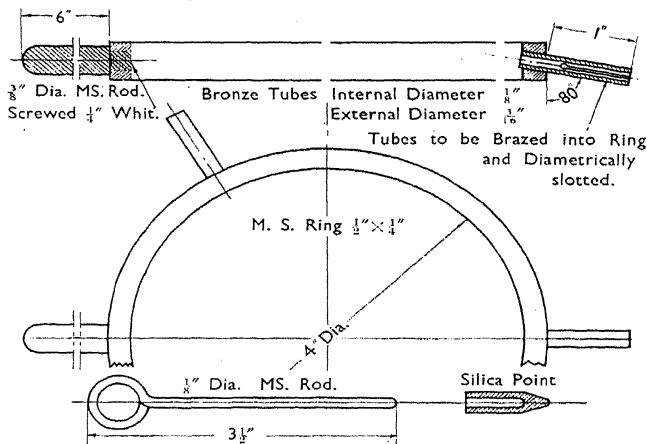


Fig. 44.—Volatile Matter in Pitch—Platinum Crucible and Lid.

A water manometer connected by means of a T piece with the gas supply entering the burner.

A retort stand and clamp to hold a three-arm crucible support with silica points (see Fig. 45, below), so arranged that when in position the bottom of the crucible is 10 mm above the burner, the crucible being completely enveloped by the flame (see Fig. 43, page 327).

A standard draught screen (Schedule No. S1, page 372).



The dimensions in the above figure are for guidance.

Fig. 45.—Volatile Matter in Pitch—Three-Arm Crucible Support with Silica Points.

Method:

The apparatus shall be assembled as indicated above, with the silica points holding the crucible as near as practicable to the rim. A small quantity of powdered pure potassium chromate shall be spread over the bottom of the crucible. The gas shall be lit with the flame fully aerated. The gas pressure at the burner shall be slowly increased until incipient fusion of the potassium chromate is apparent. The final manometer reading shall be noted as corresponding to the gas pressure to be used in the test on the pitch. The lid of the crucible shall not be used during the trials.

The crucible shall be cleaned and weighed without the lid. One gram of the finely divided sample shall be weighed into the crucible which, with the lid fitted, shall be placed in

position as before. The gas shall be relit and the gas pressure readjusted if necessary until the manometer reading is the same as that noted in the preliminary trial. The burner shall be placed in position under the crucible and after three minutes, during which the manometer reading shall be kept constant, the flame shall be extinguished. The crucible shall be allowed to cool and shall be weighed without the lid. The loss in weight represents the volatile matter in the amount of sample taken for test; the percentage by weight in the sample shall be calculated.

If the method has been properly performed, any deposit on the lid at the end of the experiment will be due to decomposition of the volatile matter. If there be any evidence that molten pitch has spurted on to the lid, the test shall be discarded and a fresh determination made.

Limits of Accuracy:

The results of repeat determinations on portions of the same sample should fall within ± 0.5 unit of the average of the several results (see page 4).

SERIAL No.: P.T. 7-38

Insoluble Matter ("Free Carbon")

Method:

One gram of the sample, finely divided if possible, shall be mixed intimately in a 200 ml beaker with 100 ml of pure toluole (B.S. 805—1938, Part A) at laboratory temperature. The particles of pitch shall be thoroughly broken up by means of a glass rod and after settlement for twenty minutes, the supernatant liquid shall be decanted cautiously, avoiding disturbance of the sediment, either (a) through balanced filter papers (e.g. Whatman No. 5) about 150 mm in diameter, or (b) through a weighed Gooch crucible prepared with asbestos and used under vacuum.

If filter papers are used, they must be heated to a temperature between 95° and 110°C and reduced to equal weight by removing the apex of the heavier paper; the uncut paper is used inside the originally heavier paper. The separated material is retained by the inner paper, but both of them are equally subjected to any action exerted by the toluole.

The residue in the beaker shall be treated in exactly the same manner with a further 100 ml of the pure toluole at

laboratory temperature. It shall then be treated with three successive quantities, each of 100 ml of hot pure toluole (see Note i) in exactly the same manner except that (a) the beaker shall stand on the water-bath during the settling periods and (b) each settling period need only be five minutes.

The residue shall be transferred to the filter and the washing continued until a further 500 ml of the hot pure toluole have been used. The residue shall be dried in an oven at a temperature between 95° and 110°C until it is of constant weight. The residue shall be incinerated, the weight of the ash deducted (after allowing for that in the filter paper) and the percentage by weight of ash-free "free carbon" shall be calculated and reported (see Note ii).

Notes:

(i) The temperature of the hot toluole must be between 90° and 100°C throughout the time it is being used.

(ii) *If the above method be varied by the use of any solvent other than pure toluole to the above mentioned specification, the result shall be reported as "Matter insoluble in ———" and not as "free carbon."*

SERIAL No.: P.T. 8-38

Ash

Method:

One gram of the sample shall be incinerated to constant weight in a platinum or porcelain crucible.

If a determination of the volatile matter has been carried out, the ash may conveniently be determined by incinerating, in the same crucible, the residue thereby obtained.

The percentage by weight of ash in the sample shall be calculated and reported.

Appendix I: Apparatus Schedules

Standard Apparatus:

Where apparatus described in the following schedules is specified for use in any test, standard apparatus is to be used. Apparatus is regarded as standard only if it complies with the relevant specification and, where such tests are prescribed, has been tested and approved at the National Physical Laboratory.

It is to be noted that the textual part of this Appendix takes precedence over the illustrations wherever there is an apparent difference between the two.

Where British Standard apparatus has been adopted, only a summary of the specification has been included in the following pages. *The relevant British Standard specifications shall be used as the basis for manufacture and testing, and not the summaries.*

STANDARD THERMOMETERS

(Schedule Nos T1a-T17a)

1. Type:

Standard S.T.P.T.C. thermometers shall be of the mercury in glass type, solid stem, and filled with inert gas at a suitable pressure.

2. Stem:

The stem shall be of lead glass or other suitable glass with enamel back. The stem shall be at least 5.5 mm but not more than 7 mm in diameter.

3. Bulb:

The bulb shall be cylindrical and in alignment with the stem. The diameter of the bulb shall not be greater than that of the stem. The bulb shall be of a thermometric glass approved by the National Physical Laboratory and suitable for the range of temperature covered (see Table 28, page 334).

4. Annealing:

The thermometer shall be suitably annealed before it is graduated.

5. Graduation and Figuring:

The distance from the bottom of the bulb to the bottom of the scale shall be at least 100 mm.

The graduation lines shall be clearly etched and of uniform thickness not exceeding 0.15 mm. The lines shall all be at right angles to the axis of the thermometer. When the thermometer is viewed from the front and in the vertical position, the lines shall all finish on a line parallel to the axis on the left hand side. Certain of the graduation lines shall be extended on the right hand side as indicated in Table 27, page 333, but the shortest graduations shall not extend across the bore.

If the diameter of the stem permits, the figures shall be upright when the thermometer is viewed from the front and in the vertical position, and should be placed so that they would be intersected by the line to which they refer if it were extended.

6. Immersion Line:

A mark, consisting of a ring carried completely round the stem of the thermometer, shall be etched on the stem at the point to which the thermometer is intended to be immersed.

This requirement shall not apply in the case of thermometer T3b, which is to be graduated for total immersion (see Table 27, opposite).

The depth of immersion shall be measured from the bottom of the bulb.

7. Safety Chamber:

A safety chamber shall be provided at the upper end of the bore and no division shall be within 10 mm of any enlargement of the bore.

8. Finish of Top of Stem:

The thermometer shall be finished with a glass ring, the diameter of which shall not exceed that of the stem.

9. Constructional Details:

Each thermometer shall comply in all respects with the details relevant to the thermometer as shewn in Table 27, opposite.

The shapes of the bulbs and expansion and contraction chambers shall be such as not to entrap small quantities of mercury or gas (as, for example, a safety chamber with a sharply pointed end or an irregularly shaped contraction chamber) and the design should be such that the mercury does not wholly recede into the main bulb at normal air temperatures.

10. Inscriptions:

Each thermometer shall have permanently and legibly marked on it:—

- (a) The letter "C" or "F," to indicate whether the Centigrade or Fahrenheit scale is employed.
- (b) The inscription "100 mm Immersion," to indicate the immersion for which the thermometer is graduated, except in the case of thermometer T3b, specimens of which shall bear the inscription "Total Immersion," to indicate that the thermometer is graduated for total immersion.
- (c) An inscription, e.g. "Nitrogen filled" to indicate the gas filling employed.
- (d) An identification number.
- (e) The maker's or vendor's name or mark.
- (f) The letters "S.T.P.T.C." and schedule mark allotted to the thermometer, e.g. "T1a," "T3a," etc., as shewn in Table 27, opposite.

The object of the inscription (f) is to indicate at once the specification with which the thermometer is intended to comply. Purchasers should not, however, accept thermometers as complying with the specification merely because they are marked in accordance with (f) as this inscription is only an identification mark and not a guarantee.

TABLE 27: CONSTRUCTIONAL DETAILS FOR STANDARD S.T.P.T.C. THERMOMETERS

Schedule mark	Range	Graduation	Longer lines at each (a)	Fully figured at each (b)	Maximum overall length	Minimum length of main scale	Maximum bulb length	Immersion	Maximum error	Maximum distance from bottom of bulb to top of contraction chamber	Limit of accuracy of N.P.L. test
		degree	degree	degree	mm	mm	mm	mm	degree	mm	degree
T 1a	-10° to 20°C	0.1	0.5 & 1	2	410	230	15	100	±0.2	(c)	±0.05
T 3a	0° to 120°C	0.5	1 & 5	10	430	240	15	100	±0.8	(c)	±0.1
T 3b	0° to 120°C	0.5	1 & 5	10	430	240	15	Total	±0.3	(c)	±0.1
T 4a	90° to 400°C	1	5	10	430	250	15	100	±4.0	25	±0.5
T 5a	15° to 45°C	0.1	0.5 & 1	2	430	255	15	100	±0.4	(c)	±0.05
T 7a	65° to 90°C	0.1	0.5 & 1	2	400	215	12	100	±0.4	25	±0.05
T 8a	70° to 130°C	0.2	1	5	380	200	15	100	±0.6	25	±0.1
T 9a	50° to 210°C	0.5	1 & 5	10	430	240	12	100	±0.8	25	±0.1
T 11a	105° to 115°C	0.1	0.5 & 1	1	300	100	12	100	±0.4	25	±0.05
T 12a	130° to 160°C	0.1	0.5 & 1	2	420	240	12	100	±0.6	25	±0.05
T 14a	150° to 250°C	0.5	1 & 5	10	390	200	15	100	±1.2	25	±0.2
T 15a	40° to 85°F	0.5	1 & 5	10	240	70	15	100	±0.4	(c)	±0.1
T 16a	175° to 230°C	0.2	1	5	430	240	15	100	±1.2	25	±0.2
T 17a	39.5° to 70.5°C	0.1	0.5 & 1	2	405	210	15	100	±0.4	25	±0.05

NOTES:

- (a) Where two numbers appear in this column, the lines corresponding to the second number shall be slightly longer than those corresponding to the first number.
- (b) The figuring of thermometers which are to be figured at each 2 degrees shall apply to the even number degrees.
- (c) These thermometers do not require a contraction chamber, but if there be any enlargement of the bore below the scale, it shall be placed entirely within a distance of 25 mm from the bottom of the bulb.
- (d) Certain dimensional requirements, common to all the thermometers, appear in clauses 2, 3 and 5 of the specification.
- (e) The limits of accuracy to be applied shall be those specified in the last column of the list of thermometers in the last column, which shall be the limit of accuracy to be applied to each thermometer submitted for test.
- (f) The requirements for thermometers T1a and T17a are such that the British Standard thermometers A30C/100 and A70C/100 of I.S. 592-1035: General Purposes Laboratory Thermometers, may be used for all tests for which thermometers T1a and T17a are respectively prescribed.

Verification

No thermometer shall be regarded as standard unless it has been certified by the National Physical Laboratory as complying with the appropriate specification. Thermometers so approved will be marked with the Laboratory monogram and year of test, and a certificate of test will be issued shewing the corrections to be applied.

It is desirable that thermometers be retested at intervals not exceeding five years, or more frequently if determinations of the zero point or other suitable fixed point indicate that a retest is required. A change of one or two divisions does not necessarily indicate the need for a complete retest, as this may be due to a normal change in the volume of the bulb and can be allowed for by applying a correction, equal to the zero or other fixed point change, throughout the scale.

Note:

Certain examples of thermometers of the 1929 series (Schedule Nos T1 to 15) may comply with the requirements for the above series (Schedule Nos T1a to 17a); such thermometers shall not be rejected by reason only of the fact that they are not marked with the new schedule marks.

TABLE 28: THERMOMETRIC GLASSES APPROVED BY THE NATIONAL
PHYSICAL LABORATORY
(AS AT NOVEMBER, 1938)

Glass	Identification mark	Approved for temperatures up to
Normal glass made by James Powell & Sons (Whitefriars), Ltd	Single blue stripe	350°C*
Normal glass made by Frank Tomey & Co., Ltd	Two blue stripes	350°C*
Normal glass, Jena 16 ^{III} , made by Schott & Genossen	Red stripe	350°C*
Normal glass, Gege Eff, made by Glaswerk Gustav Fischer	Blue and red stripes	350°C
Normal glass, Dial, made by Plowden & Thompson Ltd	Double blue stripe	350°C
Corning boro-silicate made by Corning Glass Co.	—	450°C
Jena 59 ^{III} made by Schott & Genossen	—	450°C
Jena 2954 ^{III} made by Schott & Genossen	Single black stripe	500°C
* May be used up to 400°C if a careful examination of the zero is made periodically.		

STANDARD HYDROMETERS

(Schedule Nos H1 and H2)

DENSITY HYDROMETERS

(Schedule No. H1)

British Standard Density Hydrometers

Where density hydrometers are specified in the tests, hydrometers conforming to B.S. 718—1936: Density Hydrometers, shall be used. The specification itself shall be the basis for the manufacture and testing of the hydrometers. The details below are intended as a guide in the selection of hydrometers suitable for the determination of the densities of tar and tar products.

1. Series of Density Hydrometers Available:

The hydrometers all have subdivisions with an average length of 1.6 mm. Three different scale lengths are provided, namely:—

<i>Mean scale length</i>	<i>Number of subdivisions</i>
160 mm	100
80 mm	50
40 mm	25

The specification also provides for three different degrees of openness of scale, namely:—

<i>Density equivalent of one subdivision</i>
0.0005 g/ml
0.001 g/ml
0.002 g/ml

There are eight series of hydrometers. Each series is designated by a combination of the density equivalent of one subdivision and the number of subdivisions in the nominal range of the hydrometer, e.g. 0.0005×100 , all hydrometers of this series thus having a range of 0.05 g/ml. Details of the British Standard density hydrometers covering the range of tar and tar products are given in Table 29, page 336.* As there shewn, a wide choice of hydrometers for use with tars and tar products is available to meet the varying requirements regarding accuracy and amounts of liquid available.

2. Basis of Scale of Density Hydrometers:

British Standard density hydrometers are adjusted to indicate density (mass per unit volume) in grams per millilitre at 20°C. Thus, when read in a liquid at 20°C, the hydrometers give the density of the liquid at 20°C in grams per millilitre.

The hydrometers are adjusted to indicate density when readings are taken at the intersection of the level liquid surface and the scale. The

* British Standard density hydrometers of the Series 0.0005×100 are similar in size and openness of scale to the S.T.P.T.C. Series A specific gravity hydrometers; those of the Series 0.001×50 are similar in size and openness of scale to the S.T.P.T.C. Series B specific gravity hydrometers; those of the Series 0.002×25 have the same openness of scale as the S.T.P.T.C. Series D specific gravity hydrometers but are rather larger.

TABLE 29: BRITISH STANDARD DENSITY HYDROMETERS AVAILABLE FOR USE WITH TAR AND TAR PRODUCTS

Series	Density equivalent of one sub-division g/ml	Maximum permissible scale error g/ml	Maximum permissible overall length of hydrometers mm	Mean scale length mm	Range of individual hydrometers in series covering range of density 0.650 to 1.300 g/ml of tar and tar products	Number of hydrometers for range 0.650 to 1.300 g/ml
0.0005 × 100	0.0005	±0.0004	360	160	0.650 to 0.700 g/ml; 0.700 to 0.750 g/ml and so on up to and including 1.250 to 1.300 g/ml	13
0.001 × 100	0.001	±0.001	350	160	0.600 to 0.700 g/ml; 0.700 to 0.800 g/ml and so on up to and including 1.200 to 1.300 g/ml	7
0.002 × 100	0.002	±0.002	340	160	0.600 to 0.800 g/ml; 0.800 to 1.000 g/ml and so on up to and including 1.200 to 1.400 g/ml	4
0.0005 × 50	0.0005	±0.0004	270	80	0.650 to 0.675 g/ml; 0.675 to 0.700 g/ml and so on up to and including 1.275 to 1.300 g/ml	26
0.001 × 50	0.001	±0.001	260	80	0.650 to 0.700 g/ml; 0.700 to 0.750 g/ml and so on up to and including 1.250 to 1.300 g/ml	13
0.002 × 50	0.002	±0.002	250	80	0.600 to 0.700 g/ml; 0.700 to 0.800 g/ml and so on up to and including 1.200 to 1.300 g/ml	7
0.001 × 25	0.001	±0.001	195	40	0.650 to 0.675 g/ml; 0.675 to 0.700 g/ml and so on up to and including 1.275 to 1.300 g/ml	26
0.002 × 25	0.002	±0.002	185	40	0.650 to 0.700 g/ml; 0.700 to 0.750 g/ml and so on up to and including 1.250 to 1.300 g/ml	13

hydrometers covering the range of density 0.650 g/ml to 1.300 g/ml are adjusted for the surface tensions given in Table 30, page 338.

3. Inscriptions on Density Hydrometers:

It is prescribed that each hydrometer shall have permanently and legibly marked on the paper bearing the scale or on a label in the bulb:—

- (a) The inscription "g/ml at 20°C" to indicate the basis of the scale.
- (b) The surface tension for which the hydrometer is adjusted, e.g. "S.T. 35 dynes/cm."
- (c) An identification number.
- (d) The maker's or vendor's name or mark.
- (e) The inscription "Max. Temp. 50°C" or "Max. Temp. 100°C."
- (f) The inscription "B.S.718."

The object of the inscription (f) is to indicate at once the number of the British Standard Specification with which the hydrometer is intended to comply. Purchasers should not, however, accept hydrometers as complying with the specification merely because they are marked in accordance with (f) as this inscription is only an identification mark and not a guarantee. The requirements under "Distinguishing Marks" on page 340 apply equally to density hydrometers.

SPECIFIC GRAVITY HYDROMETERS

(Schedule No. H2)

General Specification:

The S.T.P.T.C. standard specific gravity hydrometers shall conform to the general specification set out below. Provision is made for four series of hydrometers covering a range in each series of 0.650 to 1.300 in thirteen separate instruments, each having a range of 0.05.

1. The hydrometer shall be made of glass and shall be as free as possible from striæ and similar defects and in particular the external surface of the stem shall be quite smooth. The glass shall be of a kind which sufficiently resists the action of chemicals and possesses properties such as would render it suitable for use for thermometers, i.e. it should have low hysteresis and small secular change in volume.

2. The hydrometer shall be thoroughly annealed before the scale is fixed.

3. The main bulb shall be cylindrical and the cross section of the stem circular.

4. A thermometer shall not be part of the instrument.

5. When mercury is used for loading, it shall be contained in a bulb at the base of the hydrometer, suitably sealed off with glass from the main bulb of the instrument.

6. In special hydrometers for use at temperatures higher than 38°C, any material used for sealing off the bulb or for holding in position loading material, such as shot, shall not soften below 100°C (see also page 41 regarding cooling and draining after use).

7. The hydrometer shall be symmetrically constructed, so that it floats with the stem vertical.

TABLE 30: SURFACE TENSIONS FOR WHICH BRITISH STANDARD DENSITY HYDROMETERS ARE ADJUSTED

Series 0.0005 \times 50 Series 0.001 \times 25	Series 0.0005 \times 100 Series 0.001 \times 50 Series 0.002 \times 25	Series 0.001 \times 100 Series 0.002 \times 50	Series 0.002 \times 100	Surface tension for which individual hydrometers are adjusted—dynes/cm
0.650 to 0.675 g/ml 0.675 to 0.700 g/ml 0.700 to 0.725 g/ml 0.725 to 0.750 g/ml	0.650 to 0.700 g/ml 0.700 to 0.750 g/ml	0.600 to 0.700 g/ml	0.600 to 0.800 g/ml	20
0.750 to 0.775 g/ml 0.775 to 0.800 g/ml 0.800 to 0.825 g/ml 0.825 to 0.850 g/ml	0.750 to 0.800 g/ml 0.800 to 0.850 g/ml	0.700 to 0.800 g/ml	0.700 to 0.900 g/ml	25
0.850 to 0.875 g/ml 0.875 to 0.900 g/ml 0.900 to 0.925 g/ml 0.925 to 0.950 g/ml	0.850 to 0.900 g/ml 0.900 to 0.950 g/ml	0.800 to 0.900 g/ml	0.800 to 1.000 g/ml	30
0.950 to 0.975 g/ml 0.975 to 1.000 g/ml and so on up to and including 1.300 g/ml	0.950 to 1.000 g/ml 1.000 to 1.050 g/ml and so on up to and including 1.250 to 1.300 g/ml	0.900 to 1.000 g/ml 1.000 to 1.100 g/ml and so on up to and including 1.200 to 1.300 g/ml	1.000 to 1.200 g/ml 1.200 to 1.400 g/ml	35*

* British Standard density hydrometers for densities greater than 1.000 g/ml may also be obtained adjusted for 75 dynes/cm, but those adjusted for 35 dynes/cm are the appropriate ones for tars and tar products.

8. The paper used for the scale shall be of high quality and shall conform to the requirements that it shall not discolour at 100°C with use and that all divisions may be sharply and accurately marked on it.

9. (a) The subdivisions of the scale shall be without evident irregularities.

(b) The graduation marks shall be fine straight lines which lie in planes perpendicular to the axis of the hydrometer, so that the graduation marks are horizontal when the stem is vertical. The scale shall be straight and without twist and fixed with a cement which will not soften at the highest temperature at which the hydrometer is to be used.

(c) The scale of each hydrometer in Series A shall be graduated at each end for four subdivisions beyond its nominal range, for two subdivisions in Series B, and for one subdivision in Series C and in Series D; i.e. the scale of each hydrometer shall exceed the nominal range (0.050) by 0.002 at each end of the scale.

(d) Sufficient of the graduation marks shall be numbered to enable the exact reading at any point to be noted easily. At least every 0.01 graduation mark, e.g. 0.650, 0.660, etc. shall be numbered. Abbreviated numbers, if used, must be confined to the central portion of the scale; the highest and lowest numbered graduation marks must be designated in full, with the decimal point inserted and preceded by the figure "0" or the figure "1" according to the range of the hydrometer. The numbers must not encroach on the space occupied by the shortest graduation marks and must not touch or intersect any graduation mark.

(e) The length of the graduation marks must be varied so that the exact reading at any mark can be noted easily. The shortest graduation marks must be at least 2 mm in length.

10. A fine horizontal line shall be etched on the stem of the hydrometer and this shall coincide with the horizontal part of a "pointing mark" which shall be ruled on the paper scale about 5 mm above the highest graduation mark. This "pointing mark" shall be a short, fine line with a V at each end, thus >---< .

11. The hydrometer shall be graduated for reading correctly at the point of intersection of the level liquid surface with the stem.

12. The hydrometer shall be adjusted so that the reading on the scale in a liquid at 15.5°C and having a surface tension equal to that for which the hydrometer is adjusted, is the specific gravity $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of the liquid.

Each hydrometer shall be adjusted for the surface tension given against the range of the hydrometer below:—

<i>Hydrometer ranges</i>	<i>Surface tension</i>
0.650 to 0.700 and 0.700 to 0.750	20 dynes/cm
0.750 to 0.800 and 0.800 to 0.850	25 dynes/cm
0.850 to 0.900 and 0.900 to 0.950	30 dynes/cm
0.950 to 1.000 and 1.000 to 1.050 and so on up to and including 1.250 to 1.300	35 dynes/cm

13. Each hydrometer shall have permanently and legibly marked on the paper bearing the scale or on a label enclosed in the bulb:—

(a) The inscription " $S_{15.5^{\circ}\text{C}/15.5^{\circ}\text{C}}$ at 15.5°C" to indicate the basis of the scale.

- (b) The surface tension for which the hydrometer is adjusted, e.g. "S.T. 30 dynes/cm".
- (c) The inscription "S.T.P.T.C." and an identification number.
- (d) The maker's or vendor's name or mark.
- (e) The inscription "Max. Temp. 100°C" or "Max. Temp. 38°C" according to whether the cementing material for fixing the loading material is or is not suitable for the hydrometer to be used above 38°C.

The object of the inscription "S.T.P.T.C." under (c) is to indicate at once the specification with which the hydrometer is intended to comply. Purchasers should not, however, accept hydrometers as complying with the specification merely because they are marked "S.T.P.T.C." as this inscription is only an identification mark and not a guarantee.

Distinguishing Marks on Standard Instruments:

No hydrometer shall be regarded as standard unless it has been tested by the National Physical Laboratory for accuracy and dimensions and certified by the National Physical Laboratory monogram being etched on the instrument.

Details of the Hydrometer Series:

The four series of hydrometers shall comply with the respective dimensional requirements shewn in Table 31, below.

TABLE 31: S.T.P.T.C. SPECIFIC GRAVITY HYDROMETERS—GENERAL

	Series A	Series B	Series C	Series D
Maximum overall length	360 mm	250 mm	195 mm	150 mm
Maximum diameter of the bulb ..	40 mm	30 mm	25 mm	20 mm
Length of the nominal } not less range corresponding } than to 0.05 change in } not more specific gravity } than	155 mm	75 mm	50 mm	35 mm
Number of subdivisions in nominal range	175 mm	90 mm	60 mm	40 mm
Minimum distance of the highest graduation mark from the top of the stem	100	50	25	25
Minimum length of the plain cylindrical stem below the lowest graduation mark.. ..	25 mm	20 mm	20 mm	20 mm
	5 mm	5 mm	5 mm	5 mm

The external diameter of the stem of the individual hydrometers shall not be less than the lower figure nor greater than the higher figure shewn in Table 32, opposite.

The tolerance of accuracy at any point for standard hydrometers in the four series shall be:—

Series A	Series B	Series C	Series D
± 0.0004	± 0.0010	± 0.0015	± 0.0020

TABLE 32: S.T.P.T.C. SPECIFIC GRAVITY HYDROMETERS—STEM DIAMETERS

Range	Series A	Series B	Series C	Series D
	mm	mm	mm	mm
0.650-0.700	7.5-8.0	8.0-8.4	6.9-7.2	5.7-5.9
0.700-0.750	7.2-7.7	7.7-8.1	6.7-7.0	5.5-5.7
0.750-0.800	6.9-7.4	7.5-7.9	6.5-6.8	5.3-5.5
0.800-0.850	6.7-7.2	7.2-7.6	6.3-6.6	5.2-5.4
0.850-0.900	6.5-7.0	7.0-7.4	6.1-6.4	5.0-5.2
0.900-0.950	6.3-6.8	6.8-7.2	5.9-6.2	4.9-5.1
0.950-1.000	6.1-6.6	6.6-7.0	5.7-6.0	4.7-4.9
1.000-1.050	6.0-6.5	6.4-6.8	5.6-5.9	4.6-4.8
1.050-1.100	5.8-6.3	6.2-6.6	5.5-5.8	4.5-4.7
1.100-1.150	5.7-6.2	6.1-6.5	5.3-5.6	4.4-4.6
1.150-1.200	5.5-6.0	6.0-6.4	5.2-5.5	4.3-4.5
1.200-1.250	5.4-5.9	5.8-6.2	5.1-5.4	4.2-4.4
1.250-1.300	5.3-5.8	5.7-6.1	5.0-5.3	4.1-4.3

STANDARD VOLUMETRIC GLASSWARE

(Schedule Nos V1 to V11)

Where British Standard apparatus has been adopted, only a summary of the specification has been included in the following pages. *The relevant British Standard specifications shall be used as the basis for manufacture and testing, and not the summaries.*

Standard procedure demands the use of apparatus fulfilling the requirements of the Class A tests of the National Physical Laboratory. It is in the best interests of buyers and sellers of tar and tar products that the volumetric glassware used in the tests, whether ordinary apparatus such as burettes, or the special apparatus described below, should have been tested at the National Physical Laboratory and approved as complying with the requirements for Class A tests.

The Class A tolerances are designed to meet the requirements of apparatus intended to possess the highest accuracy. The Class B tolerances represent a standard for ordinary grade apparatus.

In the internal routine work of the industry, everyday works' practice may not demand a greater accuracy than is obtainable by the use of volumetric glassware fulfilling the requirements of the Class B tests.

CROW RECEIVERS

(Schedule Nos V1, V2 and V3)

100 ml British Standard Crow Receiver (Schedule No. V1)

50 ml British Standard Crow Receiver (Schedule No. V2)

25 ml British Standard Crow Receiver (Schedule No. V3)

The receivers shall comply with B.S. 605—1935: Crow Receivers. Both stoppered and unstoppered receivers are available; the unstoppered forms are illustrated in Figs 46, 47 and 48, page 342.

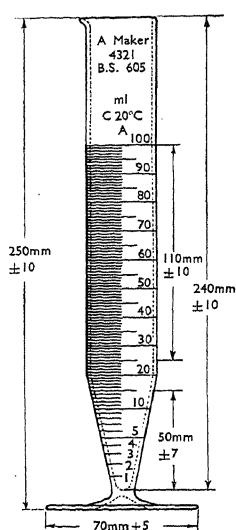


Fig. 46.

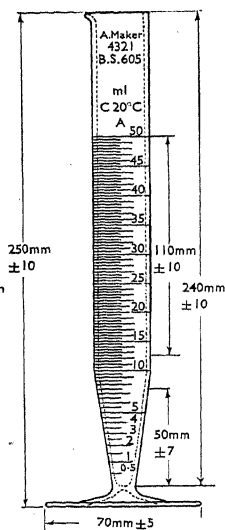


Fig. 47.

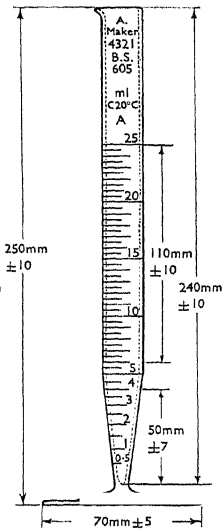


Fig. 48.

100 ml, 50 ml and 25 ml Crow Receivers (Schedule Nos VI, V2 and V3).

TABLE 33: TOLERANCE ON CAPACITY OF BRITISH STANDARD CROW RECEIVERS

Class of tolerance	V1: 100 ml receiver		V2: 50 ml receiver		V3: 25 ml receiver	
	Up to and including 10 ml mark	Above 10 ml mark	Up to and including 5 ml mark	Above 5 ml mark	Up to and including 2.5 ml mark	Above 2.5 ml mark
Class A tolerance	ml ±0.2	ml ±0.4	ml ±0.1	ml ±0.2	ml ±0.04	ml ±0.1
Class B tolerance	ml ±0.4	ml ±0.8	ml ±0.2	ml ±0.4	ml ±0.1	ml ±0.2

The tolerances on capacity of the receivers are shown in Table 33, above.

The tolerances on the lower portions of the receivers represent the maximum permissible error at any point tested on these portions and also the maximum permissible difference between the errors at any two

points tested on these portions. Thus, for example, the portion up to and including 10 ml on the 100 ml receiver may be in error by ± 0.2 ml for Class A (± 0.4 ml for Class B) at any point provided that the difference between the errors at any two points tested on this portion does not exceed 0.2 ml for Class A (0.4 ml for Class B).

The tolerances on the upper portions of the receivers represent the maximum permissible error at any point tested on these portions and also the maximum permissible difference between the error at any point on these portions and that at any other point on the receiver. Thus, for example, the portion above the 10 ml mark on the 100 ml receiver may be in error by ± 0.4 ml for Class A (± 0.8 ml for Class B) at any point provided that the difference between the error at any point tested on this portion of the receiver and that at any other point on the receiver does not exceed 0.4 ml for Class A (0.8 ml for Class B).

Receivers intended to comply with the Class B tolerances only may, if desired, have graduation marks confined to the front of the receiver.

SEPARATING FUNNEL RECEIVERS

(Schedule Nos V4 and V4a)

1. Type:

The separating funnel receivers are illustrated in Figs 49 and 50, page 344; each consists of a pear-shaped bulb at the upper end of which is a cylindrical neck and at the lower end a graduated tube fitted with a stopcock at the bottom.

2. Sizes:

Two separating funnel receivers are provided, one with a graduated portion of total capacity 3 ml subdivided into intervals of 0.05 ml (Schedule No. V4) and one with a graduated portion of total capacity 1 ml subdivided into intervals of 0.02 ml (Schedule No. V4a).

3. Material:

The separating funnel receivers shall be made of glass as free as possible from striæ and similar defects and shall be well annealed.

4. Construction:

(a) The axes of the neck and the graduated tube shall lie in a straight line with the axis of symmetry of the bulb.

(b) The top of the neck shall be slightly flared and, if desired, fitted with a ground glass stopper.

(c) The delivery tube below the stopcock shall be made of thick-walled tubing. The delivery jet shall be made with a gradual taper, the taper at the extreme end being slight so that there is no sudden constriction at the orifice. The end of the jet shall be ground off smooth and square with the axis of the jet and slightly bevelled.

(d) The stopcock shall adequately prevent leakage when the key of the stopcock is in the shut off position.

5. Dimensions:

The separating funnel receivers shall comply with the dimensions given in Table 34, page 345.

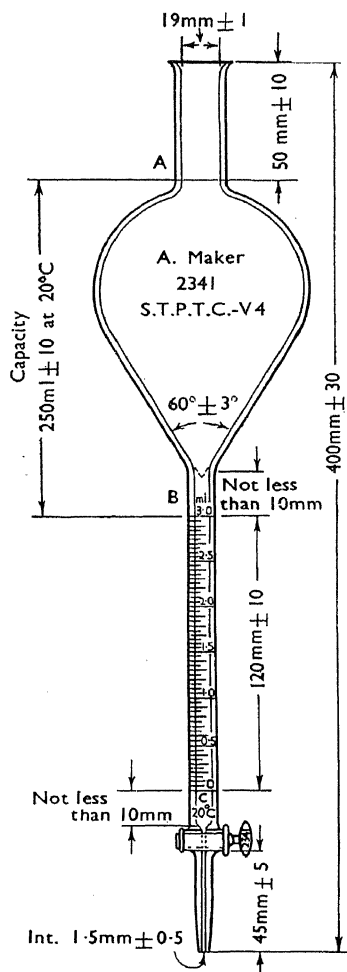


Fig. 49.—Separating Funnel Receiver (Schedule No. V 4).

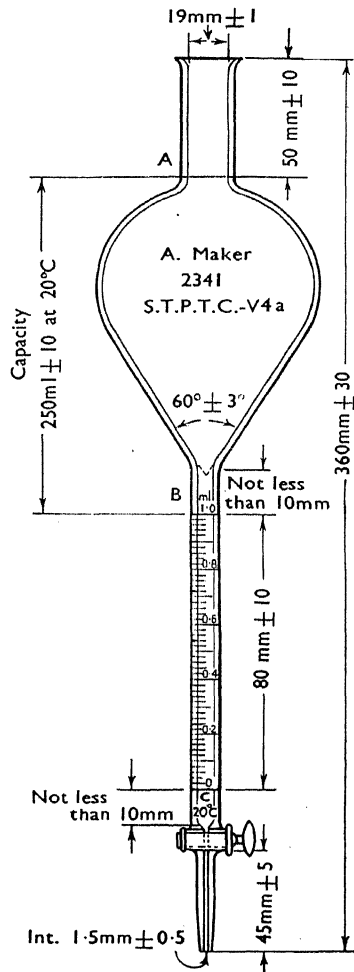


Fig. 50.—Separating Funnel Receiver (Schedule No. V 4a).

TABLE 34: DIMENSIONS OF STANDARD SEPARATING FUNNEL RECEIVERS

<i>Dimensions</i>		<i>Separating Funnel Receiver</i>	
		<i>with 3 ml scale (Schedule No. V4)</i>	<i>with 1 ml scale (Schedule No. V4a)</i>
(a) Overall length	mm	400 \pm 30	360 \pm 30
(b) Internal diameter of neck	mm	19.0 \pm 1.0	19.0 \pm 1.0
(c) Length of neck*	mm	50 \pm 10	50 \pm 10
(d) Cone angle of lower conical portion of bulb		60° \pm 3°	60° \pm 3°
(e) Minimum length of tube of uniform diameter above highest graduation mark	mm	10	10
(f) Distance between highest and lowest graduation marks	mm	120 \pm 10	80 \pm 10
(g) Minimum length of tube of uniform diameter below lowest graduation mark	mm	10	10
(h) Length of delivery tube below stop-cock	mm	45 \pm 5	45 \pm 5
(i) Internal diameter of orifice of jet ..	mm	1.5 \pm 0.5	1.5 \pm 0.5

* Dimension (c) is measured from the top of the neck to the point at which the neck begins to expand at its junction with the bulb.

6. Capacity of Bulb:

The capacity of the bulb is defined by the volume at 20°C of the water required to fill the separating funnel receiver from the highest graduation mark to the base of the neck at 20°C. The capacity so defined shall be 250 ml \pm 10.

7. Definition of Capacity of Graduated Portion:

The separating funnel being first filled to the lowest graduation mark with water at 20°C, the capacity corresponding to any graduation mark is defined by the volume in millilitres of water at 20°C which must be added in order to bring the water surface up to the graduation mark in question, the lowest point of the meniscus being set on each mark in turn and the axis of the graduated tube being vertical.

8. Graduation Marks:

The graduation marks shall be fine, cleanly etched, permanent lines of uniform thickness and shall lie in planes perpendicular to the axis of the graduated tube.

9. Numbering of Graduation Marks:

The lowest graduation mark, i.e. that nearest to the stopcock, shall be numbered "0" and the scales shall be numbered as follows:—

<i>Schedule No.</i>	<i>Total capacity of graduated portion</i>	<i>Numbered graduation marks</i>
V4	3 ml	0, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 ml
V4a	1 ml	0, 0.2, 0.4, 0.6, 0.8 and 1.0 ml

10. Length of Graduation Marks:

(a) *Separating funnel receiver with 3 ml scale—Schedule No. V4:* The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.05 ml, 0.15 ml, 0.25 ml and so on up to and including 2.95 ml shall be carried half-way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks.

(b) *Separating funnel receiver with 1 ml scale—Schedule No. V4a:* The numbered graduation marks shall be carried completely round the tube, the graduation marks midway between the numbered marks shall be carried about three-quarters the way round the tube and the remaining graduation marks half the way round the tube. The graduation marks midway between the numbered marks shall extend equally at each end beyond the shortest graduation marks.

11. Position of Graduation Marks and Numbers:

When the separating funnel is vertical with the stopcock at the bottom and the handle of the stopcock on the right, the ends of the shortest graduation marks shall lie vertically beneath each other down the centre of the front and the back of the graduated tube, the marks extending to the left from front to back. The numbers shall then appear upright on the front of the graduated tube, slightly to the right of the ends of the short graduation marks and immediately above the graduation marks to which they relate.

Scales subdivided and numbered in accordance with Clauses 9 to 11 are shown in Figs 49 and 50.

12. Tolerances on Capacity of Graduated Portion:

The tolerances on the capacity of the graduated portion as defined in Clause 7 are:—

Class of tolerance	Separating Funnel Receiver	
	with 3 ml scale (Schedule No. V4)	with 1 ml scale (Schedule No. V4a)
	ml	ml
Class A tolerance	± 0.03	± 0.01
Class B tolerance	± 0.05	± 0.02

The tolerances represent the maximum error allowed at any point tested and also the maximum permissible difference between the errors at any two points tested. Thus, for example, the 3 ml scale of the separating funnel receiver Schedule No. V4 may be in error by ± 0.03 ml for Class A (± 0.05 ml for Class B) at any point provided that the difference between the errors at any two points tested does not exceed 0.03 ml for Class A (0.05 ml for Class B).

13. Alternative Graduation of Class B Separating Funnel Receivers:

Graduated separating funnel receivers intended to comply with the Class B tolerances only may, if desired, have graduation marks confined to the front of the graduated tube instead of graduation marks as specified in Clause 10. The length of these graduation marks must be suitably varied so as to emphasize the same graduation marks as are emphasized by the provisions of Clause 10.

14. Inscriptions:

Each separating funnel receiver shall have permanently and legibly marked on it:—

- (a) The abbreviation "ml" to indicate that the apparatus is calibrated in terms of the millilitre. This inscription shall be placed between the highest graduation mark and the bulb.
- (b) The inscription "C. 20°C" to indicate that the apparatus is calibrated for content at 20°C.
- (c) An identification number, repeated on the handle of the stopcock and on the ground glass stopper when one is fitted at the neck.
- (d) The maker's or vendor's name or mark.
- (e) The inscription "S.T.P.T.C.—V4" on separating funnel receivers with a 3 ml graduated portion and "S.T.P.T.C.—V4a" on separating funnel receivers with a 1 ml graduated portion, to indicate that the receivers are intended to comply with this specification; and "A" or "B" according to the class of accuracy.

The object of the inscription (e) is to indicate at once the specification with which the separating funnel receiver is intended to comply. Purchasers should not, however, accept units as complying with the specification merely because they are marked in accordance with (e) as this inscription is only an identification mark and not a guarantee.

PHENOLS FLASKS

(Schedule Nos V5 and V5a)

British Standard Flasks with Graduated Necks

The phenols flasks are illustrated in Figs 51 and 52, page 348, and shall comply respectively with the requirements for (a) the flask with a 200 ml bulb and 25 ml scale and (b) the flask with a 150 ml bulb and 10 ml scale in B.S. 676—1936: Three Special Flasks with Graduated Necks.

The capacity of the bulb, i.e. the volume of water required to fill the flask to the 0 ml graduation mark at 20°C, the lowest point of the water meniscus being set on the mark, is 200 ml \pm 10 in the case of flask Schedule No. V5 and 150 ml \pm 7 in the case of flask Schedule No. V5a.

The tolerances on the graduated portions of the flasks are:—

	200 ml flask with 25 ml scale (Schedule No. V5)		150 ml flask with 10 ml scale (Schedule No. V5a)	
	Class A	Class B	Class A	Class B
Maximum permissible error on capacity corresponding to any graduation mark ..	± 0.1	± 0.2	± 0.04	± 0.08
Maximum permissible difference between the errors at any two graduation marks ..	0.1	0.2	0.04	0.08

Flasks intended to comply with the Class B tolerances only may, if desired, have graduation marks confined to the front of the neck.

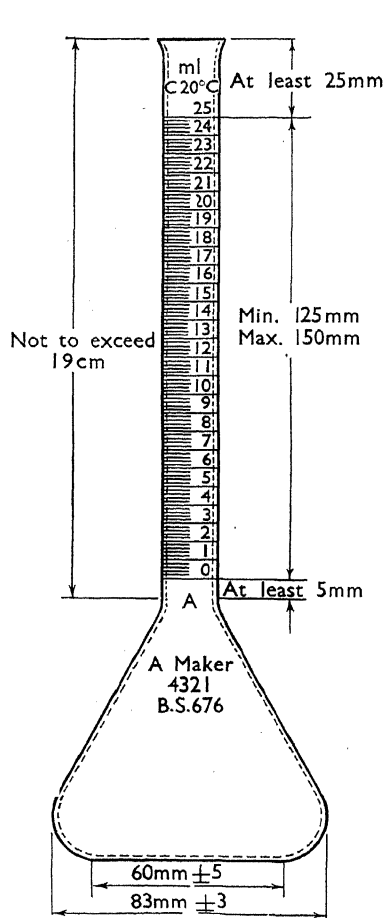


Fig. 51.—Phenols Flask
(Schedule No. V5).

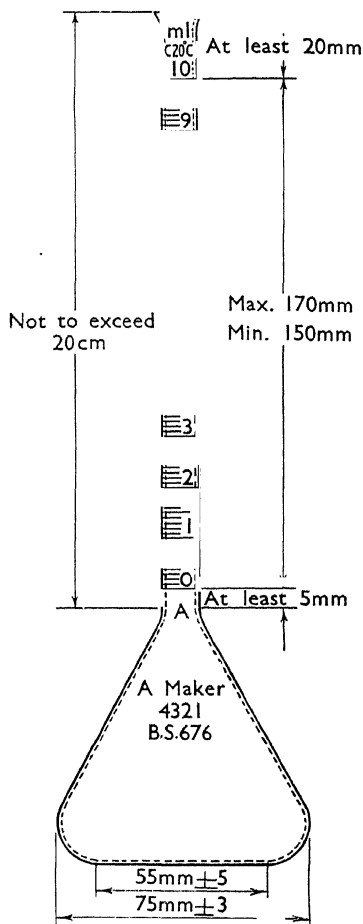


Fig. 52.—Phenols Flask
(Schedule No. V5a).

DEAN AND STARK APPARATUS

(Schedule Nos V6 and V6a)

10 ml and 2 ml Standard Apparatus for the Determination of Water by Distillation with an Immiscible Liquid*

The following particulars are based on a British Standard Specification at present in draft form. The specification will be published as B.S. 614—1938 or 1939 and when it is published, must be regarded as superseding this schedule except in respect of the flask to be used; this shall be in accordance with the instructions in the relevant test and, so far as they apply, the requirements of Clause 2 below.

The British Standard Specification will be based primarily on the use of ground glass joints which in any event are obligatory with the 2 ml receiver. The specification will provide also for cork connexions in the case of the 10 ml receiver and the alternative form may be used at the discretion of the operator when testing tar or tar products.

Any order for this apparatus must indicate which form—2 ml or 10 ml receiver, ground joints or cork connexions, etc.—is required wherever alternatives are provided below.

1. Assembly of Apparatus:

The assembly of the apparatus when a 2 ml receiver is employed is shewn in Fig. 53, page 350, and in this assembly the 10 ml receiver having ground glass joints can be substituted for the 2 ml receiver.

When the 10 ml receiver not fitted with ground glass joints is used, a condenser and flask suitable for assembly by means of corks and otherwise complying with the following specification (see Clauses 2 and 3) shall be used.

In addition to the three components flask, receiver and condenser shewn in Fig. 53, a spray tube is provided (see Clause 4) for the removal of droplets of water from the inner tube of the condenser by means of a spray of the immiscible liquid.

The component parts of the apparatus shewn in Fig. 53 and the alternative receivers and spray tube shall comply with the requirements of the following clauses.

2. Flask:

A glass or metal flask, which may be flat or round bottomed at the discretion of the operator, will be required, depending on the instructions under "Apparatus" in the particular test. If a glass flask be there specified, it shall comply with the following requirements:—

(a) *Material*: The flask shall be made of hard resistance glass, shall be as free as possible from striæ and similar defects and shall be well annealed.

(b) *Finish*: Except when required for use with a 10 ml receiver without ground glass joints, the top of the neck shall be ground to form the socket of a standard A29 ground glass joint.

(c) *Capacity*: The flask shall have a nominal capacity of 500 ml.

* (i) Frequently called entrainment distillation.

(ii) The 10 ml and 2 ml sizes of the Type 1 receivers of the British Standard series are alone required in the testing of tar and its products.

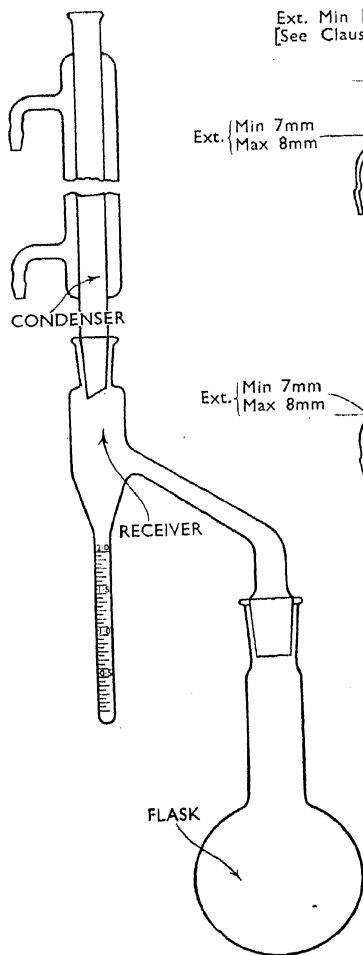


Fig. 53.—Assembly with 2 ml Receiver.

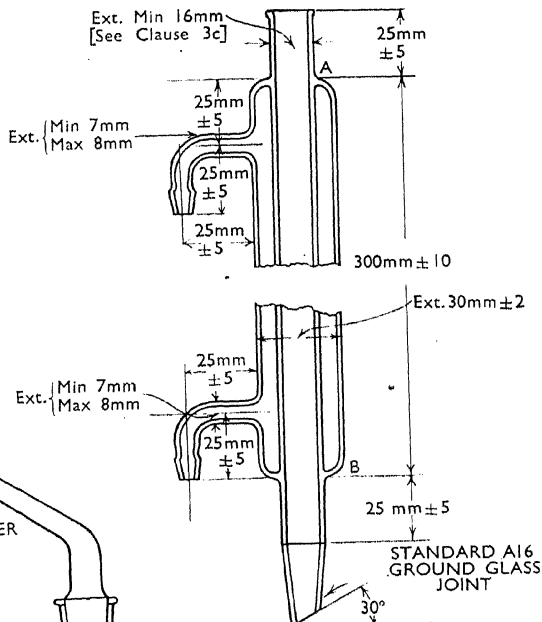


Fig. 54.—Condenser.

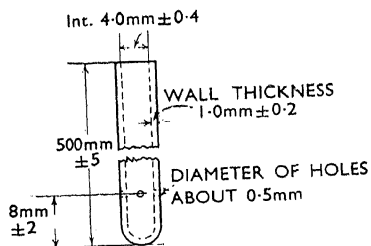


Fig. 55.—Spray Tube.

3. Condenser:

(a) *Material:* The condenser shall be made of hard resistance glass, shall be as free as possible from striæ and similar defects and shall be well annealed.

(b) *Construction and Dimensions:* The condenser shall be constructed as shewn in Fig. 54, opposite, and shall conform to the dimensions and tolerances given in Fig. 54.

(c) *Central Tube of Condenser:* Except when required for use with a 10 ml receiver without ground glass joints, the lower end of the central tube shall terminate in the cone of a standard A16 ground glass joint, but with the lower end ground off at an angle of about 30° as shewn in Fig. 54.

The external diameter of the central tube shall be about but not less than 16.0 mm, so that the junction between the cylindrical part of the tube and the conical joint will not restrict the free flow of liquid down the inside wall of the central tube. The central tube shall be of the thinnest wall tubing compatible with forming a ground glass joint at the end.

(d) *Length of Condenser:* The length of the central portion of the condenser AB of Fig. 54 shall be 300 mm \pm 10.

4. Spray Tube:

The spray tube is shewn in Fig. 55, opposite, and shall be a glass tube sealed at one end and having four small holes, spaced equidistantly round the wall of the tube at a short distance from the closed end of the tube. The tube shall conform to the following dimensions:—

Overall length	500 mm \pm 5
Internal diameter	4.0 mm \pm 0.4
Wall thickness	1.0 mm \pm 0.2
Diameter of holes	about 0.5 mm
Distance of centres of holes from sealed end of tube	8 mm \pm 2

5. 10 ml Receiver (Schedule No. V6):

(a) *Material:* The receiver shall be made of hard resistance glass, shall be as free as possible from striæ and similar defects and shall be well annealed.

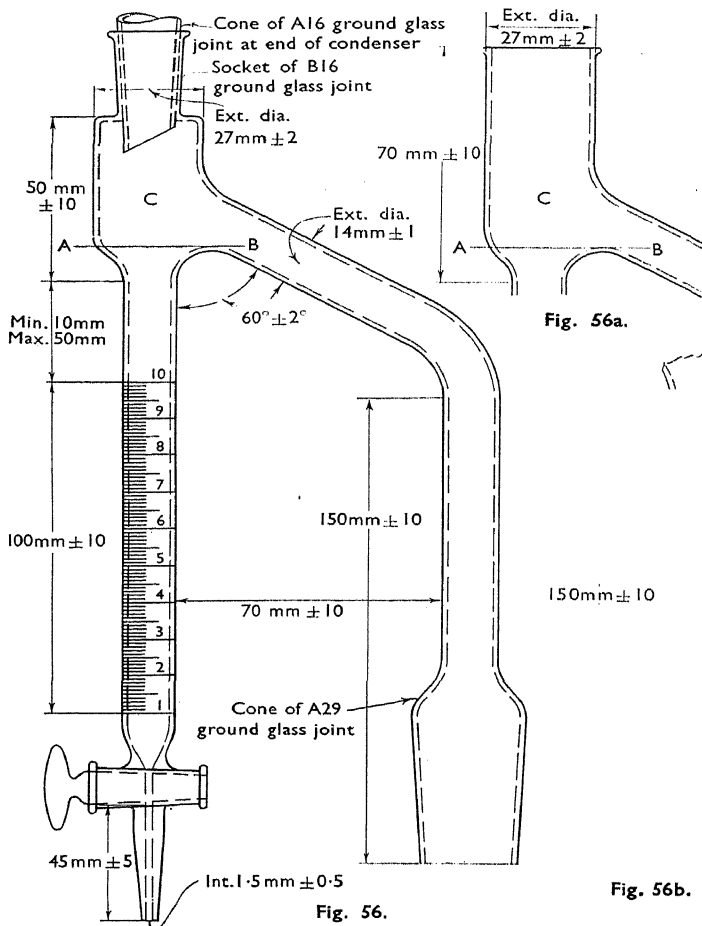
(b) *Dimensions:* The 10 ml receiver fitted with ground glass joints shall conform to the dimensions and tolerances shewn in Fig. 56, page 352 and that without ground glass joints shall conform to the dimensions and tolerances shewn in Fig. 56 as modified by Figs 56a and 56b, page 352.

(c) *Upper Chamber:* The upper chamber C in Fig. 56 shall have a neck ground to form the socket of a standard B16 ground glass joint. The shoulder immediately below the neck shall be finished square as shewn in Fig. 56.

The overflow level AB shall be approximately at the base of the chamber C in both types of receiver (see Figs 56 and 56a).

(d) *Finish of Bottom of Graduated Tube:* A stopcock with a delivery jet below it shall be sealed to the bottom of the graduated tube.

The stopcock shall satisfactorily prevent leakage when the key is in the shut-off position.



Inscriptions to appear on receiver as required by Clause 5 (n) of specification.

Fig. 56.—10 ml Receiver (Schedule No. V6) with Ground Glass Joints.

Fig. 56a.—Design of Upper Chamber without Ground Glass Socket.

Fig. 56b.—Design of Side Tube without Ground Glass Cone.

Dean and Stark Apparatus

The delivery jet shall be made with a gradual taper with no sudden constriction at the orifice. The end of the jet shall be ground off smooth and square with the axis of the jet. The internal diameter of the orifice at the tip of the jet shall be $1.5 \text{ mm} \pm 0.5$.

(e) *Capacity of Graduated Portion:* The graduated portion shall have a capacity of 10 ml at 20°C when filled to the highest graduation mark. The capacity corresponding to any graduation mark is defined by the volume in millilitres of water at 20°C required to fill the graduated portion up to that mark at 20°C , the stopcock being closed, the axis of the graduated tube being vertical and the lowest point of the water meniscus being set on the graduation mark. The volume of the hole through the key of the stopcock and the volume of the jet below the stopcock shall not be counted as part of the measuring space.

(f) *Range and Subdivision of Scale:* The scale shall cover the range 1 ml to 10 ml and shall be subdivided into intervals of 0.1 ml.

(g) *Length of Graduation Marks:* The numbered graduation marks shall be carried completely round the tube. The graduation marks midway between the numbered marks shall be carried about three-quarters the way round the tube and the remaining graduation marks shall be carried half-way round the tube. The graduation marks midway between the numbered marks shall project equally at each end beyond the shortest graduation marks.

(h) *Graduation Marks:* The graduation marks shall be fine, cleanly etched, permanent lines of uniform thickness and shall lie in planes perpendicular to the axis of the graduated tube.

(j) *Numbering of Graduation Marks:* The graduation marks corresponding to 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml, 7 ml, 8 ml, 9 ml and 10 ml shall be numbered.

(k) *Position of Scale and Numbers:* The graduation marks shall be confined to the cylindrical portion of the tube. The tube shall also be cylindrical for not less than 10 mm and not more than 50 mm above the highest graduation mark.

When the receiver is in the position shewn in Fig. 56, the ends of the shortest graduation marks shall lie vertically beneath each other down the centre of the front and of the back of the tube, the marks extending to the left from front to back. The numbers shall then appear upright on the front of the tube immediately above the graduation marks to which they relate and to the right of the ends of the graduation marks level with the numbers.

The scheme of graduation and numbering specified in Clauses 5 (f) to 5 (k) is illustrated in Fig. 56.

(l) *Length of Scale:* The distance between the 1 ml and 10 ml graduation marks shall be $100 \text{ mm} \pm 10$.

(m) *Tolerance on Capacity:* The error at any point on the graduated scale shall not exceed $\pm 0.06 \text{ ml}$ and the difference between the errors at any two points on the scale shall not exceed 0.06 ml.

(n) *Inscriptions:* Each receiver shall have permanently and legibly marked on it:—

- (i) The abbreviation "ml" to signify that the receiver is calibrated in terms of the millilitre.
- (ii) The inscription " $^{\circ}\text{C. } 20^{\circ}\text{C}$ " to indicate that the receiver is calibrated for content at 20°C .

- (iii) An identification number. The identification number shall be marked both on the receiver and on the handle of the stopcock.
- (iv) The maker's or vendor's name or mark.
- (v) The inscription "S.T.P.T.C.—V6" and "A" or "B" according to the class of accuracy.

Note:

The object of the inscription (v) is to indicate at once the specification with which the receiver is intended to comply. Purchasers should not, however, accept receivers as complying with the specification merely because they are marked in accordance with (v) as this inscription is only an identification mark and not a guarantee.

6. 2 ml Receiver (Schedule No. V6a):

(a) *Material:* The receiver shall be made of hard resistance glass, as free as possible from striæ and similar defects, and well annealed.

(b) *Dimensions:* The receiver shall conform to the dimensions and tolerances shewn in Fig. 57, opposite.

(c) *Upper Chamber:* The upper chamber A in Fig. 57 shall have a neck ground to form the socket of a standard B16 interchangeable ground-glass joint. The shoulder immediately below the neck shall be finished square as shewn in Fig. 57.

(d) *Finish of Bottom of Graduated Tube:* The bottom of the graduated tube shall be sealed, the end of the tube being approximately hemispherical in shape. The seal shall be such that the graduated tube remains cylindrical down to at least the 0.1 ml graduation mark.

(e) *Capacity of Graduated Portion:* The graduated portion shall have a capacity of 2 ml at 20°C when filled to the highest graduation mark. The capacity corresponding to any graduation mark is defined by the volume in millilitres of water at 20°C required to fill the graduated portion to that mark at 20°C, the axis of the graduated tube being vertical and the lowest point of the water meniscus being set on the graduation mark.

(f) *Range and Subdivision of Scale:* The scale shall cover the range 0.1 ml to 2 ml and shall be divided into intervals of 0.05 ml.

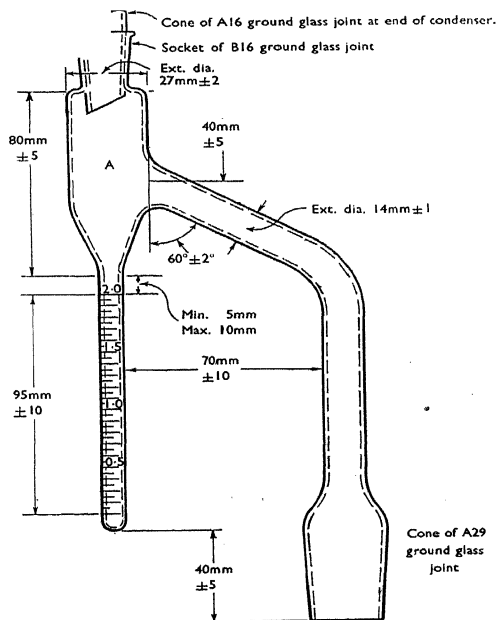
(g) *Length of Graduation Marks:* The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.15 ml, 0.25 ml, 0.35 ml and so on up to and including 1.95 ml, shall be carried half-way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks.

(h) *Graduation Marks:* The graduation marks shall be fine, cleanly etched, permanent lines of uniform thickness and shall lie in planes perpendicular to the axis of the graduated tube.

(j) *Numbering of Graduation Marks:* The graduation marks corresponding to 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml shall be numbered.

(k) *Position of Scale and Numbers:* The graduated tube shall remain cylindrical for not less than 5 mm and not more than 10 mm above the highest graduation mark.

When the receiver is in the position shewn in Fig. 57, the ends of the shortest graduation marks shall lie vertically beneath each other down the centre of the front and of the back of the tube, the marks extending



Inscriptions to appear on receiver as required by Clause 6 (n) of specification.

Fig. 57.—Dean and Stark Apparatus—2 ml Receiver (Schedule No. V6a).

to the left from front to back. The numbers shall then appear upright on the front of the tube, immediately above the graduation marks to which they relate and to the right of the graduation marks level with the numbers.

The scheme of graduation and numbering specified in Clauses 6 (f) to 6 (k) is illustrated in Fig. 57.

(l) *Length of Scale*: The distance between the 0.1 ml and 2 ml graduation marks shall be $95 \text{ mm} \pm 10$.

(m) *Tolerance on Capacity*: The error at any point on the scale shall not exceed $\pm 0.03 \text{ ml}$ and the difference between the errors at any two points shall not exceed 0.03 ml .

(n) *Inscriptions*: Each receiver shall have permanently and legibly marked on it:—

- (i) The abbreviation “ml” to signify that the receiver is calibrated in terms of the millilitre,

- (ii) The inscription "C. 20°C" to indicate that the receiver is calibrated for content at 20°C.
- (iii) An identification number.
- (iv) The maker's or vendor's name or mark.
- (v) The inscription "S.T.P.T.C.—V6a" and "A" or "B" according to the class of accuracy.

The object of the inscription (v) is to indicate at once the specification with which the receiver is intended to comply. Purchasers should not, however, accept receivers as complying with the specification merely because they are marked in accordance with (v), as this inscription is only an identification mark and not a guarantee.

Sulphonation Apparatus (Schedule Nos V7a and V7b)

1. Type:

The sulphonation apparatus is illustrated in Figs 58 and 59, opposite, and consists of a spherical bulb connected by a graduated tube to a smaller pear-shaped bulb. The larger bulb is fitted with a ground glass stopper in line with the graduated tube and a stopcock is fitted at the opposite end of the apparatus.

2. Sizes:

Sulphonation apparatus of two sizes are provided, one having a graduated portion of total capacity 10 ml and subdivided into intervals of 0.1 ml (Schedule No. V7a) and one having a graduated portion of total capacity 2 ml and subdivided into intervals of 0.05 ml (Schedule No. V7b).

3. Material:

The apparatus shall be made of glass as free as possible from striæ and similar defects and shall be well annealed.

4. Construction:

(a) The centres of the bulbs shall be in line with the axis of the tube which shall pass centrally through the stopper and the stopcock.

(b) The stopper shall be a good fit and prevent leakage when liquid is shaken in the larger bulb. A stopper of suitable size is one in which the ground portions of the stopper and of the neck form the cone and socket respectively of the size B19 joint in B.S. 572—1934: Interchangeable Conical Ground Glass Joints.

(c) The stopcock shall satisfactorily prevent leakage when the key of the stopcock is in the shut-off position.

(d) The extension below the stopcock shall be of thick-walled tubing and its lower end shall be finished smooth and square with the axis of the tube.

5. Dimensions:

The apparatus shall comply with the dimensions shewn in Table 35, page 358.

6. Capacity of Larger Bulb:

The apparatus being first filled to the graduation mark nearest to the larger bulb with water at 20°C, the lowest point of the water surface

lowest point of the water surface being set on this mark and the axis of the graduated tube being vertical. The capacity so defined shall be 90 ml \pm 2 for Schedule No. V7a and 98 ml \pm 2 for Schedule No. V7b.

8. Definition of Capacity of Graduated Portion:

The apparatus being first filled to the lowest graduation mark with water at 20°C, the capacity corresponding to any graduation mark is defined by the volume in millilitres of water at 20°C which must be added in order to bring the water surface up to the graduation mark in question, the lowest point of the meniscus being set on each mark in turn and the axis of the graduated tube being vertical.

TABLE 35: DIMENSIONS OF SULPHONATION APPARATUS

<i>Dimensions</i>	<i>Sulphonation Apparatus</i>	
	<i>with</i> 10 ml scale (Schedule No. V7a)	<i>with</i> 2 ml scale (Schedule No. V7b)
	mm	mm
(a) Maximum overall length with stopper removed	400	400
(b) Length of tube of uniform diameter between larger bulb and graduation mark nearest to that bulb	15 \pm 5	15 \pm 5
(c) Distance between highest and lowest graduation marks	110 \pm 10	110 \pm 10
(d) Length of tube of uniform diameter between smaller bulb and graduation mark nearest to that bulb	10 \pm 5	10 \pm 5
(e) Length of uniform tube between smaller bulb and stopcock	15 \pm 5	15 \pm 5
(f) Length of tube below stopcock	15 \pm 5	15 \pm 5
(g) External diameter of graduated tube	—	9 \pm 0.5
(h) External diameter of tube between smaller bulb and stopcock	9 \pm 1	9 \pm 1
(i) Internal diameter of tube below stopcock	3 \pm 0.5	3 \pm 0.5

9. Graduation Marks:

The graduation marks shall be fine, cleanly etched, permanent lines of uniform thickness and shall lie in planes perpendicular to the axis of the graduated tube.

10. Numbering of Graduation Marks:

The lowest graduation mark, i.e. that nearest to the smaller bulb, shall be numbered "0" and the scale shall be numbered in accordance with the following table:—

<i>Total capacity of graduated portion</i>	<i>Numbered graduation marks</i>
10 ml	0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 ml
2 ml	0, 0.5, 1.0, 1.5 and 2.0 ml

11. Length of Graduation Marks:

(a) *Sulphonation Apparatus with 10 ml Scale*—Schedule No. V7a: The numbered graduation marks shall be carried completely round the tube, the graduation marks midway between the numbered marks shall be carried about three-quarters the way round the tube and the remaining graduation marks half-way round the tube. The graduation marks midway between the numbered marks shall extend equally at each end beyond the shortest graduation marks.

(b) *Sulphonation Apparatus with 2 ml Scale*—Schedule No. V7b: The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.05 ml, 0.15 ml, 0.25 ml, and so on up to and including 1.95 ml shall be carried half-way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks.

12. Position of Graduation Marks and Numbers:

When the apparatus is vertical with the stopcock at the bottom and the handle of the stopcock on the right, the ends of the shortest graduation marks shall lie vertically beneath each other down the centre of the front and the back of the graduated tube, the marks extending to the left from front to back. The numbers shall then appear upright on the front of the graduated tube, immediately above the graduation marks to which they relate and slightly to the right of the ends of the graduation marks on the front of the tube level with the numbers.

Sections of the scales subdivided and numbered in accordance with Clauses 10 to 12 are shewn in the insets to Figs 58 and 59, page 357.

13. Tolerances on Capacity of Graduated Portion:

The tolerances on the capacity of the graduated portion as defined in Clause 8 are:—

Class of tolerance	<i>Sulphonation Apparatus</i>	
	<i>with 10 ml scale</i> (Schedule No. V7a)	<i>with 2 ml scale</i> (Schedule No. V7b)
	ml	ml
Class A tolerance	± 0.06	± 0.02
Class B tolerance	± 0.10	± 0.04

The tolerances represent the maximum error allowed at any point tested and also the maximum permissible difference between the errors at any two points tested. Thus, for example, the 10 ml scale of the sulphonation apparatus Schedule No. V7a may be in error by ± 0.06 ml for Class A (± 0.10 ml for Class B) at any point provided that the difference between the errors at any two points tested does not exceed 0.06 ml for Class A (0.10 ml for Class B).

14. Alternative Graduation of Class B Sulphonation Apparatus:

Sulphonation apparatus intended to comply with the Class B tolerances only may, if desired, have graduation marks confined to the front of the graduated tube instead of graduation marks as specified in Clause 11. The length of these graduation marks must be suitably varied so as to emphasize the same graduation marks as are emphasized by the provisions of Clause 11.

15. Inscriptions:

Each sulphonation apparatus shall have permanently and legibly marked on it:—

- (a) The abbreviation "ml" to indicate that the apparatus is calibrated in terms of the millilitre.
- (b) The inscription "C. 20°C" to indicate that the apparatus is calibrated for content at 20°C.
- (c) An identification number, repeated on the handle of the stopcock and on the ground glass stopper.
- (d) The maker's or vendor's name or mark.
- (e) The inscription "S.T.P.T.C.—V7a" when the total capacity of the graduated portion is 10 ml, "S.T.P.T.C.—V7b" when the total capacity of the graduated portion is 2 ml, to indicate that the apparatus is intended to comply with this specification; and "A" or "B" according to the class of accuracy.

The object of the inscription (e) is to indicate at once the specification with which the sulphonation apparatus is intended to comply. Purchasers should not, however, accept units as complying with the specification merely because they are marked in accordance with (e) as this inscription is only an identification mark and not a guarantee.

PHENOLS SEPARATING APPARATUS

(Schedule No. V8)

1. Type:

The phenols separating apparatus is illustrated in Fig. 60, opposite, and consists of a pear shaped bulb connected by a graduated tube to a smaller elongated bulb. The larger bulb is fitted with a ground glass stopper in line with the graduated tube and a stopcock is fitted at the opposite end of the apparatus.

2. Material:

The apparatus shall be made of glass as free as possible from striæ and similar defects and shall be well annealed.

3. Construction:

(a) The centres of the bulbs shall be in line with the axis of the tube which shall pass centrally through the stopper and the stopcock.

(b) The stopper shall be a good fit and prevent leakage when liquid is shaken in the larger bulb. A stopper of suitable size is one in which the ground portions of the stopper and of the neck form the cone and socket respectively of the size B19 joint in B.S. 572—1934: Inter-changeable Conical Ground Glass Joints.

(c) The stopcock shall satisfactorily prevent leakage when the key of the stopcock is in the shut-off position.

(d) The delivery tube below the stopcock shall be made of thick-walled tubing. The delivery jet shall be made with a gradual taper, the taper at the extreme end being slight so that there is no sudden constriction at the orifice. The end of the jet shall be ground off smooth and square with the axis of the jet and slightly bevelled.

4. Dimensions:

The apparatus shall comply with the dimensions shown in Table 36, page 362.

5. Capacity of Larger Bulb:

The apparatus being first filled to the graduation mark nearest to the larger bulb with water at 20°C, the lowest point of the water surface being set on the mark, the capacity of the larger bulb is defined by the volume in millilitres of water at 20°C which must be added to bring the water surface into a position such that the lowest point of the water surface is in the plane of the bottom of the neck, the apparatus being vertical and the stopcock closed throughout. The capacity so defined shall be $140 \text{ ml} \pm 10$.

6. Definition of Capacity of Graduated Portion:

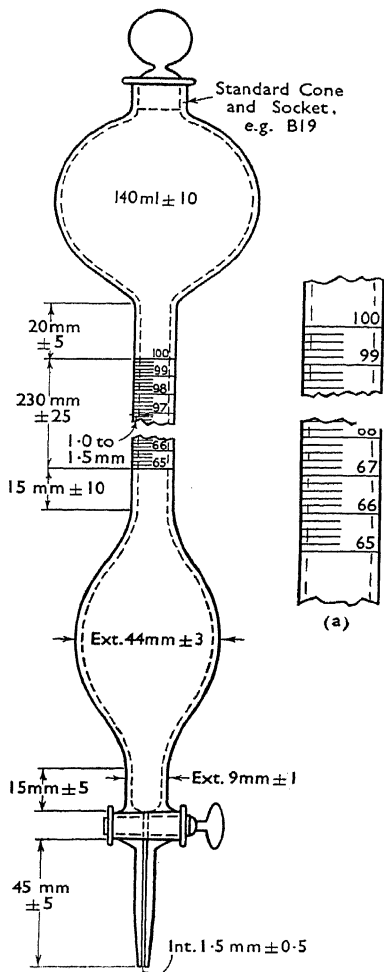
The stopcock being closed, the apparatus empty and with its axis vertical and the larger bulb uppermost, the capacity corresponding to any graduation mark is defined by the volume in millilitres of water at 20°C which must be added to bring the water surface up to the graduation mark in question, the lowest point of the meniscus being set on the graduation mark.

7. Graduation Marks:

The graduation marks shall be fine, cleanly etched, permanent lines of uniform thickness and shall lie in planes perpendicular to the axis of the graduated tube.

8. Range and Subdivision of Scale:

The range of the scale shall be from 65 ml to 100 ml and the scale shall be subdivided into intervals of 0.2 ml.



Inscriptions to appear on apparatus as required by Clause 14 of specification.

Fig. 60.—Phenols Separating Apparatus (Schedule No. V8). (Inset (a): Details of Graduation Markings.)

9. Numbering of Graduation Marks:

The lowest graduation mark, i.e. that nearest to the smaller bulb, shall be numbered "65" and the scale shall be numbered at 66 ml, 67 ml and so on up to and including 100 ml.

10. Length of Graduation Marks:

The numbered graduation marks shall be carried completely round the tube; the remaining marks shall be carried half-way round the tube.

TABLE 36: DIMENSIONS OF PHENOLS SEPARATING APPARATUS

<i>Dimensions</i>		<i>Limits</i>
		mm
(a)	Length of tube of uniform diameter between larger bulb and 100 ml graduation mark	20 \pm 5
(b)	Distance between 100 ml and 65 ml graduation marks	230 \pm 25
(c)	Wall thickness of graduated tube	1.0 to 1.5
(d)	Length of uniform tube between 65 ml mark and smaller bulb	15 \pm 10
(e)	External diameter of smaller bulb (see Fig. 60)	44 \pm 3
(f)	Length of uniform tube between smaller bulb and stopcock	15 \pm 5
(g)	Length of delivery tube below stopcock	45 \pm 5
(h)	External diameter of tube between smaller bulb and stopcock	9 \pm 1
(i)	Internal diameter of orifice of jet	1.5 \pm 0.5

11. Position of Graduation Marks and Numbers:

When the apparatus is vertical with the stopcock at the bottom and the handle of the stopcock on the right, the ends of the shortest graduation marks shall lie vertically beneath each other down the centre of the front and the back of the graduated tube, the marks extending to the left from front to back.

The numbers shall appear upright on the front of the tube, immediately above the marks to which they refer and slightly to the right of the ends of the marks level with the numbers.

The section of the scale shewn in Fig. 60a, page 361, is subdivided and numbered in accordance with Clauses 7 to 11.

12. Tolerances on Capacity of Graduated Portion:

The Class A tolerance on the capacity of the graduated portion as defined in Clause 6 is ± 0.1 ml and the Class B tolerance is ± 0.2 ml. The tolerances represent the maximum error allowed at any point tested and also the maximum permissible difference between the errors at any two points tested. Thus, for example, the scale may be in error by ± 0.1 ml for Class A (± 0.2 ml for Class B) at any point provided that the difference between the errors at any two points tested does not exceed 0.1 ml for Class A (0.2 ml for Class B).

13. Alternative Graduation of Class B Phenols Separating Apparatus:

Phenols separating apparatus intended to comply with the Class B tolerances only may, if desired, have graduation marks confined to the

front of the graduated tube instead of graduation marks as specified in Clause 10. The length of these graduation marks must be suitably varied so as to emphasize the same graduation marks as are emphasized by the provisions of Clause 10.

14. Inscriptions:

Each apparatus shall have permanently and legibly marked on it:—

- (a) The abbreviation "ml" to indicate that the apparatus is calibrated in terms of the millilitre.
- (b) The inscription "C. 20°C" to indicate that the apparatus is calibrated for content at 20°C.
- (c) An identification number, repeated on the handle of the stopcock and on the ground glass stopper.
- (d) The maker's or vendor's name or mark.
- (e) The inscription "S.T.P.T.C.—V8" to indicate that the apparatus is intended to comply with this specification and "A" or "B" according to the class of accuracy.

The object of the inscription (e) is to indicate at once the specification with which the phenols separating apparatus is intended to comply. Purchasers should not, however, accept units as complying with the specification merely because they are marked in accordance with (e) as this inscription is only an identification mark and not a guarantee.

NESSLER CYLINDERS

(Schedule Nos V9 and V10)

100 ml British Standard Nessler Cylinder (Schedule No. V9)

50 ml British Standard Nessler Cylinder (Schedule No. V10)

The cylinders shall comply with B.S. 612—1935: Nessler Cylinders, and are illustrated in Fig. 61, page 364.

The maximum permissible errors in capacity are shewn below:—

<i>Size of cylinder</i>	<i>Maximum permissible error in</i>
100 ml	± 0.8 ml at the 100 ml mark
100 ml	± 0.8 ml at the 50 ml mark
50 ml	± 0.4 ml at the 50 ml mark

On a 100 ml cylinder the difference between the error at the 100 ml mark and that at the 50 ml mark shall not exceed 0.8 ml.

DENSITY BOTTLES

(Schedule No. V11)

10 ml, 25 ml, 50 ml and 100 ml British Standard Density Bottles

The bottles shall comply with B.S. 733—1937: Density Bottles, and the 50 ml bottle is shewn, as an example of the series, in Fig. 62, page 364.

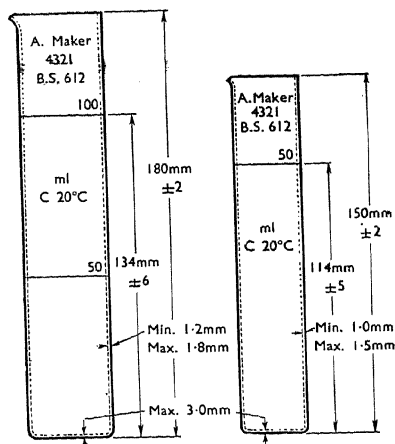


Fig. 61.—Nessler Cylinders (Schedule Nos V9 and V10).

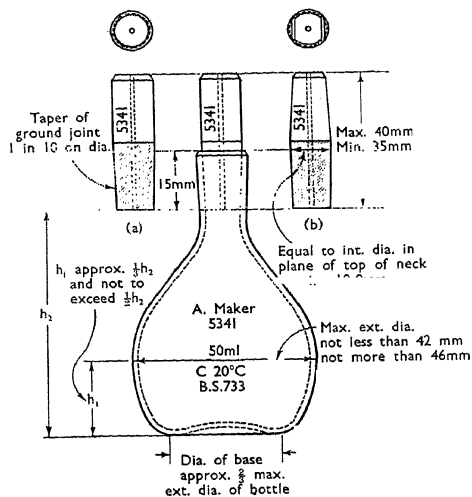


Fig. 62.—Density Bottles (Schedule No. VII).

The figure illustrates the specified dimensions of the bottle and of the alternative stoppers, but the profile of the bottle is approximate only and not intended as a precise drawing.

The recommended dimensions of the necks of the bottles, the tolerances on the capacities of the bottles and the diameters of the capillaries through the stoppers are shewn in Table 37, below.

TABLE 37: CERTAIN DIMENSIONS AND TOLERANCES ON CAPACITIES OF BRITISH STANDARD DENSITY BOTTLES

	Nominal Capacity of Bottle			
	10 ml	25 ml	50 ml	100 ml
Internal diameter in plane of top of neck* mm	7.5	7.5	10.0	12.5
Length of engagement between stopper and neck* .. mm	12	12	15	16
Taper of ground portion* ..	1: 10 on diameter, corresponding to a semi-cone angle of 2°51'45" on the stopper			
Maximum permissible error on nominal capacity ±ml	0.004	0.006	0.008	0.012
Diameter of capillary through stopper:				
Minimum mm	0.5	0.5	0.5	0.5
Maximum mm	1.0	1.0	1.5	1.5

* The dimensions quoted above are the same as the corresponding dimensions of the ground joints Nos C7, C10 and C12 respectively of B.S. 572—1934: Interchangeable Conical Ground Glass Joints.

STANDARD LABORATORY GLASSWARE

(Schedule Nos F1 to F9, B1, C1 to C3, A1 and A2)

DISTILLATION FLASKS

(Schedule Nos F1 to F9)

British Standard Distillation Flasks

The standard distillation flasks specified in the 1929 edition of "Standard Methods for Testing Tar and its Products" were taken by the British Standards Institution as the basis for the series of flasks specified in B.S. 571—1934: Distillation Flasks. The flasks are designed for use with thermometers calibrated for 100 mm immersion. The capacities and dimensions of the sizes of British Standard distillation flasks required for the standard tests are shewn in Table 38, page 367. The table shews the modifications necessary in the lengths of the side tubes in certain sizes of flasks, for compliance with the Committee's requirements. The reference letters in the second column of the table relate to the key diagram shewn in Fig. 63, page 366.

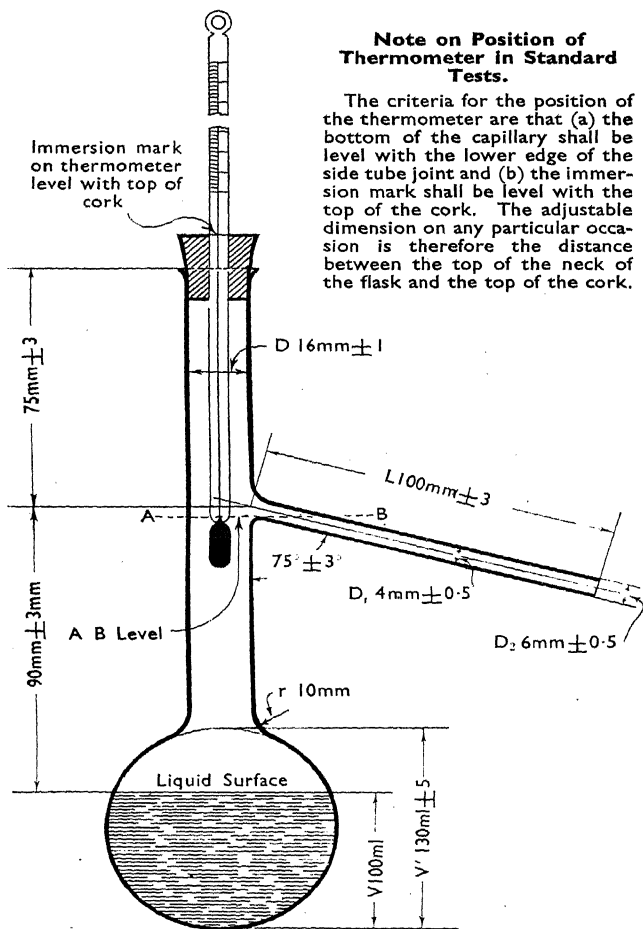


Fig. 63.—Distillation Flask (Schedule No. F2) of 100 ml Distillation Capacity, with 100 mm Immersion Thermometer in position as required by Standard Tests.

In addition to the requirements given in Table 38, the following requirements apply to each size of flask:—

Position of side tube: The distance from the centre of the side tube of each flask, at the junction with the neck, to the top of the neck shall be $75 \text{ mm} \pm 3$. The distance from the centre of the side tube of each flask, at the junction with the neck, to the liquid surface, when the flask is vertical and contains a quantity of liquid equal in volume to the distillation capacity of the flask, shall be $90 \text{ mm} \pm 3$.

Inclination of side tube: The side tube shall slope downwards from the junction with the neck so that the acute angle between the side tube and the neck is $75^\circ \pm 3^\circ$.

TABLE 38: CAPACITIES AND DIMENSIONS OF THE BRITISH STANDARD DISTILLATION FLASKS WITH DETAILS OF CHANGES IN LENGTHS OF SIDE TUBES REQUIRED FOR THE STANDARD TESTS

S.T.P.T.C. Schedule Number	F1	F2	F3	F4	F5	F6	F7	F8	F9
Distillation capacity (i)	V ml	50	100	180	250	350	500	750	1000
Capacity of bulb	V ^a ml	65	130	170	195	325	455	650	975
Tolerance		± 3	± 5	± 6	± 7	± 10	± 12	± 15	± 25
Internal diameter of neck between side arm and bulb	D mm	16	16	18	18	20	22	24	28
Tolerance		± 1	± 1	± 1	± 1	± 1	± 1	± 1	± 1
Internal diameter of side tube	D _s mm	4	4	5	5	6	6.5	7.5	8.5
Tolerance		± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5
External diameter of side tube	D _e mm	6	6	7	7	8	9	10	11
Tolerance		± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5	± 0.5
S.T.P.T.C. Length of side tube (ii)	L mm	100	100	120	120	124 ± 4	140 ± 4	140 ± 4	160 ± 5
Tolerance B.S. 571		± 3	± 3	± 4	± 4	160 ± 5	180 ± 5	200 ± 5	210 ± 6
Radius of curvature at base of neck (iii)	r mm	10	10	10	10	10	12	12	12
Thickness of walls of bulb, neck and side tube	mm	0.8 to 1.2					1.0 to 1.5		

NOTES:

(i) The distillation capacity is the maximum volume of liquid which the bulb of the flask can satisfactorily accommodate for distillation.

(ii) For use in the standard tests it is necessary that the five largest British Standard distillation flasks in the above table should have the lengths of side arms shown in heavy type.

(iii) It is desirable that the radius of curvature at the base of the neck should not be too small and so make a sharp angle between the neck and the bulb; on the other hand, the radius should not be so large as to make the bulb appreciably "pear-shaped." The radii given are for the guidance of manufacturers, but no rigid tolerances have been fixed.

STANDARD BOILING-POINT FLASK

(Schedule No. B1)

The apparatus shall be as shown in Fig. 64, page 368, and in its construction a British Standard distillation flask of 100 ml distillation capacity (see Schedule No. F2, above) shall be used.

A piece of thin walled glass tubing (wall thickness about 0.5 mm) shall be sealed to the inside of the neck of the flask. This tube shall have a hole about 6 mm in diameter blown in its side and midway between the ends. The tube shall be sealed into the neck of the flask by means of projecting points of glass so that the hole in the tube is

STANDARD METHODS

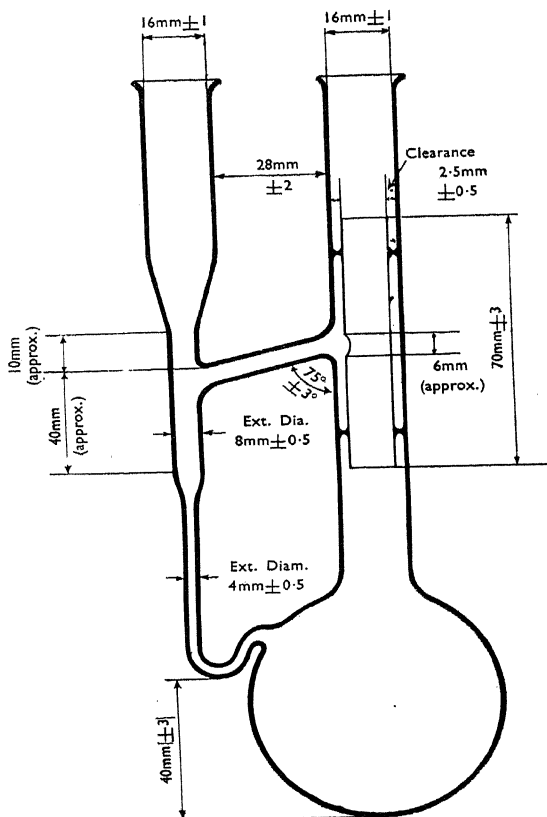


Fig. 64.—Boiling-point Flask (Schedule No. B1).

opposite the point where the side tube of the distillation flask opens into the neck of the flask. The necessary dimensions and tolerances are shown in Fig. 64, above.

The side tube of the distillation flask shall be cut off short and sealed to a vertical tube, the axis of which lies in the same vertical plane as that of the neck of the distillation flask and the side tube of the distillation flask. The vertical tube is in three coaxial sections of different diameters and the bottom section is bent into a semi-circle at its lower end and sealed to the bulb of the flask. The necessary dimensions and tolerances are shown in Fig. 64, above; the thickness of the walls of the vertical side tube should be about 1 mm.

STANDARD CONDENSERS

(Schedule Nos C1, C2 and C3)

LIEBIG CONDENSER WITH BENT END

(Schedule No. C1)

The condenser (type 1 of B.S. 658—1936) shall be of good quality resistance glass. The inner tube shall be bent at one end as shewn in Fig. 65, below, and shall conform to the following dimensions:—

Internal diameter	14 mm \pm 1.0
Wall thickness	1.0 mm to 1.5 mm
Length of straight portion of longer limb	600 mm \pm 10
Length of shorter limb (see Fig. 65)	450 mm \pm 10
Angle included between longer and shorter limb	97° \pm 3°

The open end of the longer limb shall be finished square with the axis of this limb and the open end of the shorter limb shall be ground at an angle of about 45° to the axis of this limb.

The water jacket shall have a central straight portion (see Fig. 65) of which the length is 450 mm \pm 10 and the external diameter 35 mm \pm 3. It shall be of the type shewn in Fig. 65 and fixed in position by rubber tubing.

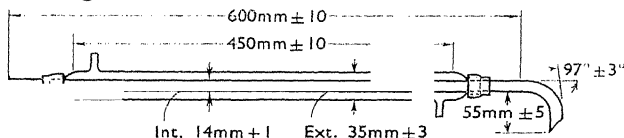


Fig. 65.—Liebig Condenser with Bent End (Schedule No. C1).

STRAIGHT LIEBIG CONDENSER

(Schedule No. C2)

The condenser (type 2 of B.S. 658—1936) is shewn in Fig. 66, below, and shall be of good quality resistance glass.

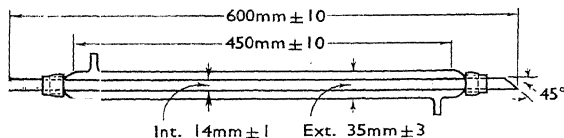


Fig. 66.—Straight Liebig Condenser (Schedule No. C2).

The inner tube shall conform to the following dimensions:—

Internal diameter	14 mm \pm 1.0
Overall length	600 mm \pm 10
Wall thickness	1.0 mm to 1.5 mm

One end of the inner tube shall be finished square with the axis and the other end shall be ground at an angle of about 45° to the axis.

The water jacket shall have a central straight portion (see Fig. 66) of which the length is $450 \text{ mm} \pm 10$ and the external diameter $35 \text{ mm} \pm 3$. It shall be of the type shewn in Fig. 66 and fixed in position by rubber tubing.

AIR CONDENSER

(Schedule No. C3)

The air condenser (type 3 of B.S. 658—1936) shall consist of a straight tube of good quality resistance glass. The tube shall conform to the following dimensions:—

Internal diameter	20 mm \pm 1.0
Overall length	600 mm \pm 10
Wall thickness	1.0 mm to 1.5 mm

One end of the tube shall be finished square with the axis and the other end shall be ground at an angle of about 45° with the axis.

STANDARD FRACTIONATING COLUMNS

(Schedule Nos A1 and A2)

1. General:

Designs for two fractionating columns are specified in this schedule, the columns being identical except in respect of the number and size of the bulbs. The Committee has considered the possibility of avoiding these differences, but such unification is impossible, having regard to commercial and technical considerations.

2. Material:

The fractionating columns shall be made of resistance glass, shall be as free as possible from striæ and similar defects and shall be well annealed.

3. Dimensions:

The fractionating columns shall have the respective dimensions shewn in Figs 67 and 68, opposite.

4. Shape of Bulbs:

The bulbs shall be of the general shape shewn in Figs 67*a* and 68*a*, and the bulbs in any column shall all be regular in shape within reasonably practical limits of manufacture.

5. Alignment:

In any fractionating column, the lower straight section of tubing, the bulbs and the upper straight section of tubing attached to the uppermost bulb shall lie concentrically about a common axis.

6. Finish:

(a) If intended for use with a cork connexion to the flask containing the material to be distilled, the open end of the lower straight section of tubing shall be ground at an angle of about 60° to the axis,

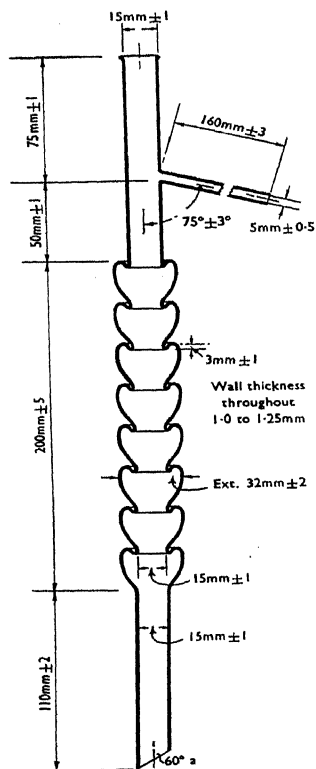


Fig. 67.—8-Bulb Fractionating Column (Schedule No. A1).

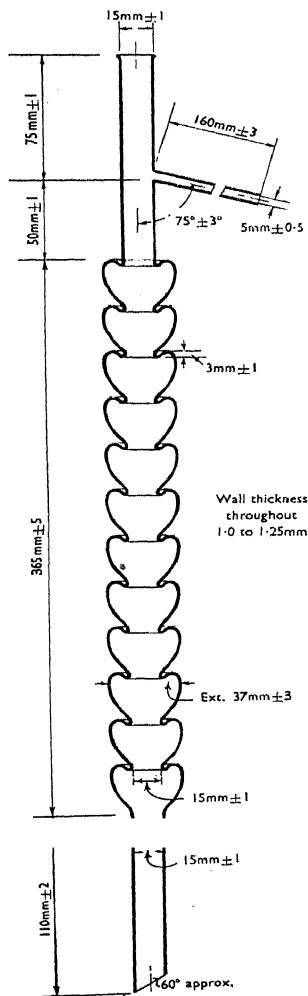


Fig. 68.—12-Bulb Fractionating Column (Schedule No. A2).

(b) If intended for use with a ground glass joint connexion to the flask containing the material to be distilled, the lower straight section of tubing shall carry a standard interchangeable ground glass joint. The distance between the bottom of the lowest bulb and the open end of the glass joint shall then be $110 \text{ mm} \pm 2$.

(c) The flange at the open end of the straight section of tubing above the uppermost bulb shall be turned over sharply and the mouth below the flange shall not be belled to any considerable distance from the flange.

7. Marking:

Each fractionating column made in accordance with this specification shall have permanently marked on it:—

- the inscription "S.T.P.T.C." and the Schedule No. "A1" or "A2" according as it is intended to comply with the specification for the 8-bulb or the 12-bulb column; and
- the maker's or vendor's name or mark.

The object of the inscription (a) is to indicate at once the specification with which the fractionating column is intended to comply. Purchasers should not however accept fractionating columns as complying with the specification merely because they are marked in accordance with (a) as this inscription is only an identification mark and not a guarantee.

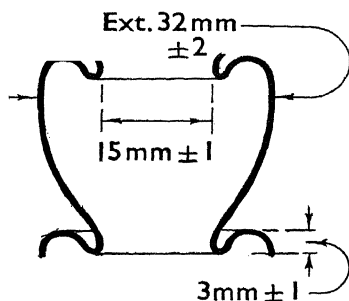


Fig. 67a.—Bulb of 8-bulb Fractionating Column (Schedule No. A1).

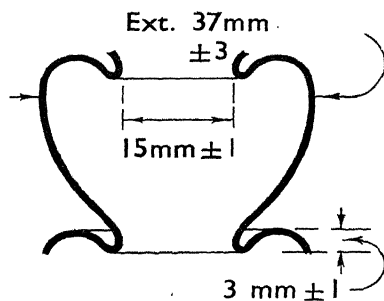


Fig. 68a.—Bulb of 12-bulb Fractionating Column (Schedule No. A2).

STANDARD DRAUGHT SCREENS

(Schedule Nos S1 to S3)

SEMI-CYLINDRICAL DRAUGHT SCREEN

(Schedule No. S1)

The standard semi-cylindrical draught screen consists of a semi-cylindrical sheet of metal, 300 mm in diameter and sufficiently high for the purpose of the test which is being carried out.

CYLINDRICAL DRAUGHT SCREEN

(Schedule No. S2)

The standard cylindrical draught screen consists of two semi-cylindrical sheets of metal hinged together at one pair of vertical edges and provided with a fastening on the other pair of vertical edges so that the two halves may be closed together to form a cylinder $300 \text{ mm} \pm 5$ in external diameter and having a height appropriate for the fractionation apparatus in conjunction with which the screen will be used. Heights of $750 \text{ mm} \pm 5$ and $1,000 \text{ mm} \pm 5$ are appropriate for screens used with the standard 8-bulb and 12-bulb pear fractionating columns respectively.

A vertical slot $30 \text{ mm} \pm 0.5$ in width and $110 \text{ mm} \pm 1$ in depth shall be formed at the top of the screen. A vertical slot about 30 mm in width and about 30 mm in height shall be formed at the bottom of the screen.

RECTANGULAR DRAUGHT SCREEN

(Schedule No. S3)

The standard rectangular draught screen shall comply with the following requirements which are identical with those for the draught screen in B.S. 658—1936: Distillation Apparatus:—

The screen shall be rectangular in cross section and open at the top and bottom. The draught screen shall be dimensioned as shewn in Fig. 69, below, and shall be made of 22 gauge sheet metal.

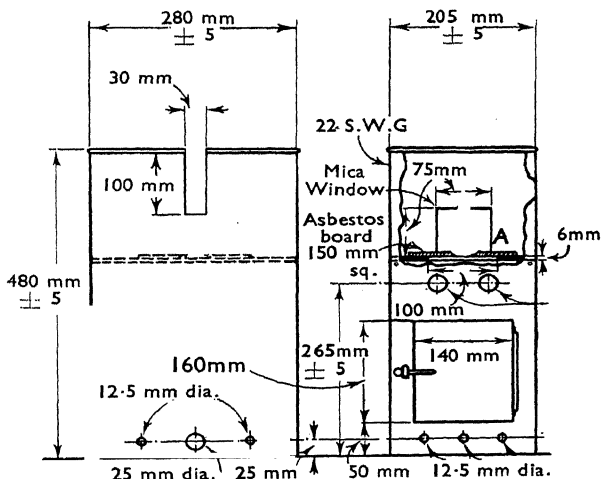


Fig. 69.—Rectangular Draught Screen
(Schedule No. S3).

In each of the two narrow sides of the draught screen there shall be two circular holes 25 mm in diameter situated below the asbestos shelf as shewn in Fig. 69. In each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the positions shewn in Fig. 69; the diameter of each of the holes centrally situated in the longer sides shall be 25 mm and the remaining ten holes shall be 12.5 mm in diameter. At the middle of each of the wider sides a vertical slot dimensioned as shewn in Fig. 69 shall be cut downwards from the top of the screen.

A sheet of hard asbestos 6 mm in thickness and having a central circular hole 100 mm in diameter shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

In one of the narrow sides of the screen a door shall be provided having the dimensions and position shewn in Fig. 69. In each of the narrow sides of the screen a mica window shall be placed centrally, with the bottom of the window level with the top of the asbestos shelf. The dimensions and position of the windows are shewn in Fig. 69.

In addition to the asbestos board referred to above, two additional asbestos boards and a sheet of wire gauze each 150 mm square are required. The additional asbestos boards shall each be 6 mm in thickness; one shall have a central hole 30 mm in diameter and the other a central hole 40 mm in diameter. The wire gauze shall have 20 mesh to the inch.

Appendix II: Physical Data

TABLE 39: ATOMIC WEIGHTS, 1938

Name	Sym- bol	Atomic Weight	Name	Sym- bol	Atomic Weight
Aluminium	Al	26.97	Neon	Ne	20.183
Antimony	Sb	121.76	Nickel	Ni	58.69
Argon	A	39.944	Niobium	Nb	92.91
Arsenic	As	74.91	(Columbium)	(Cb)	
Barium	Ba	137.36	Nitrogen	N	14.008
Beryllium	Be	9.02	Osmium	Os	190.2
Bismuth	Bi	209.00	Oxygen	O	16.0000
Boron	B	10.82	Palladium	Pd	106.7
Bromine	Br	79.916	Phosphorus	P	31.02
Cadmium	Cd	112.41	Platinum	Pt	195.23
Cæsium	Cs	132.91	Potassium	K	39.096
Calcium	Ca	40.08	Praseodymium	Pr	140.92
Carbon	C	12.010	Protoactinium	Pa	231
Cerium	Ce	140.13	Radium	Ra	226.05
Chlorine	Cl	35.457	Radon	Rn	222
Chromium	Cr	52.01	Rhenium	Re	186.31
Cobalt	Co	58.94	Rhodium	Rh	102.91
Copper	Cu	63.57	Rubidium	Rb	85.48
Dysprosium	Dy	162.46	Ruthenium	Ru	101.7
Erbium	Er	167.2	Samarium	Sm	150.43
Europium	Eu	152.0	Scandium	Sc	45.10
Fluorine	F	19.00	Selenium	Se	78.96
Gadolinium	Gd	156.9	Silicon	Si	28.06
Gallium	Ga	69.72	Silver	Ag	107.880
Germanium	Ge	72.60	Sodium	Na	22.997
Gold	Au	197.2	Strontium	Sr	87.63
Hafnium	Hf	178.6	Sulphur	S	32.06
Helium	He	4.003	Tantalum	Ta	180.88
Holmium	Ho	163.5	Tellurium	Te	127.61
Hydrogen	H	1.0081	Terbium	Tb	159.2
Indium	In	114.76	Thallium	Tl	204.39
Iodine	I	126.92	Thorium	Th	232.12
Iridium	Ir	193.1	Thulium	Tm	169.4
Iron	Fe	55.84	Tin	Sn	118.70
Krypton	Kr	83.7	Titanium	Ti	47.90
Lanthanum	La	138.92	Tungsten	W	183.92
Lead	Pb	207.21	Uranium	U	238.07
Lithium	Li	6.940	Vanadium	V	50.95
Lutecium	Lu	175.0	Xenon	Xe	131.3
Magnesium	Mg	24.32	Ytterbium	Yb	173.04
Manganese	Mn	54.93	Yttrium	Y	88.92
Mercury	Hg	200.61	Zinc	Zn	65.38
Molybdenum	Mo	95.95	Zirconium	Zr	91.22
Neodymium	Nd	144.27			

THE MILLILITRE

The kilogram was originally defined as the mass of a quantity of water which, at its temperature of maximum density, occupied a volume of one cubic decimetre. The litre could then be defined either as a cubic decimetre or as the volume, at its temperature of maximum density, of one kilogram of water.

A standard kilogram weight was prepared; the accuracy of which was subsequently doubted; the new and present standard (the International Prototype Kilogram) has the same mass as the original, but is not recognized as conforming to the original definition.

In 1901 the litre was re-defined (*vide Trav. et Mem. XII, 1920*) as the volume occupied by one kilogram of pure water at its maximum density and subjected to normal atmospheric pressure. This definition of the litre consequently bears no direct relation to the units of length, and the relation between the litre and the cubic decimetre rests solely on experimental evidence. The relation accepted at present* is:—

$$1 \text{ litre} = 1000.028 \text{ cm}^3$$

and the error is regarded as probably not exceeding 0.001 cm^3 .

The difference between the cubic centimetre and the millilitre, or thousandth part of a litre is therefore so small as to be negligible for ordinary volumetric glassware. The term cubic centimetre has, however, been extensively misapplied to denote the volume of a quantity of water having an apparent weight in air of 1 gram. In view of this confusion, and for other reasons, the Joint Committee for the Standardisation of Scientific Glassware recommended in 1924:—

“That the recognized international metric units—the litre (l) and millilitre or thousandth part of the litre (ml)—shall be used as the standard units of volume, and that standard volumetric glassware shall be graduated in terms of these units and marked ml instead of c.c.”

This recommendation has been followed extensively by British manufacturers of volumetric glassware.

* *Vide*: Ch. Ed. Guillaume: “La Création du Bureau International des Poids et Mesures” (Gauthier Villars, Paris (1927)).

THE GALLON

Imperial:

The imperial gallon is defined in Section 15 of the Weights and Measures Act, 1878 as "containing ten imperial standard pounds weight of distilled water weighed in air against brass weights, with the water and the air at the temperature of sixty-two degrees of Fahrenheit's thermometer, and with the barometer at thirty inches."

Table 40, below, shews for a few commonly used reference temperatures (*a*) the volume at the reference temperature of the quantity of water which occupies 1 gallon at 62°F, and (*b*) the weight of the quantity which occupies one gallon at the reference temperature.

TABLE 40: THE GALLON

<i>Temperature t°</i>	<i>Volume at t° of the quantity of water which occupies 1 gallon at 62°F</i>	<i>Weight in air of the quantity of water which occupies 1 gallon at t°</i>
15°C (59°F)	0.999 73 gal	10.002 7 lb
15.5°C (60°F)	0.999 82 "	10.001 8 "
16.7°C (62°F)	1.000 00 "	10.000 0 "
20°C (68°F)	1.000 63 "	9.993 7 "

United States of America:

The Gallon (U.S.A.) is defined as follows: "A Gallon (gal) is a unit of capacity equivalent to the volume of 231 cubic inches" (Bureau of Standards Circular No. 47, page 9, 1914). In evaluating this unit, the inch (U.S.A.) is taken by the Bureau of Standards from the relation 1 metre = 39.37 inches precisely (Act of Congress of 1866); this relation gives a value slightly greater than the imperial inch.

TABLE 41: STANDARD WEIGHTS AND

The following table is part of one in "Measurement of Oil in Bulk, Part I: Standard Weights and Measures," published by the Institute of Petroleum and containing much valuable information on the subject. The contracted ratios shewn below have found ready acceptance since they were recommended

	Relation						Contracted ratio
<i>Linear measures:—</i>							
METRE: yard	1.09361
" : foot	3.28084
" : inch	39.37
YARD: metre	0.9144
FOOT: metre	0.3048
INCH: millimetre	25.4
U.S. INCH: Imperial inch	1
<i>Cubic Measures:—</i>							
CUBIC METRE: cubic yard	1.30796
" " : cubic foot	35.3148
CUBIC CENTIMETRE: cubic inch	0.061024
CUBIC YARD: cubic metre	0.76455
CUBIC FOOT: cubic metre	0.0283167
CUBIC INCH: cubic centimetre	16.387
U.S.: Imperial	1.00001
IMPERIAL: U.S.	0.99999
<i>Weight:—</i>							
KILOGRAM: pound weight	2.20462
TONNE: ton (2,240 lb)	0.98421
" : "short" ton (2,000 lb)	1.10231
POUND WEIGHT: kilogram	0.453592
TON (2,240 lb): tonne	1.01605
"SHORT" TON— (2,000 lb): tonne	0.907185
U.S.: Imperial	1

MEASURES—INTER-RELATION OF UNITS

by the Institute for adoption in the petroleum industries in 1932 and they are hereby recommended for use in the tar and tar products industry.

The contracted values agree in every case with the true values, i.e. with the values calculated from the latest available experimental data, to within about one part or less in 100,000.

<i>Relation</i>						<i>Contracted ratio</i>
<i>Measures of Capacity and Cubic Measures:—</i>						
LITRE: cubic metre	0-00100003
" : cubic foot	0-0353158
" : cubic inch	61-0258
CUBIC METRE: litre	999-97
CUBIC FOOT: litre	28-316
CUBIC INCH: litre	0-0163865
IMPERIAL GALLON: litre	4-54596
" : cubic metre	0-0045461
" : cubic foot	0-160544
" : cubic inch	277-42
LITRE: Imperial gallon	0-219975
CUBIC METRE: Imperial gallon	219-97
CUBIC FOOT: Imperial gallon	6-2288
CUBIC INCH: Imperial gallon	0-00360463
U.S. GALLON: litre	3-78533
" : cubic metre	0-00378543
" : cubic foot	0-133681
" : cubic inch	231-
LITRE: U.S. gallon	0-264178
CUBIC METRE: U.S. gallon	264-17
CUBIC FOOT: U.S. gallon	7-4805
CUBIC INCH: U.S. gallon	0-004329
U.S. GALLON: Imperial gallon	0-83268
IMPERIAL GALLON: U.S. gallon	1-20094

TABLE 42: PHYSICAL CONSTANTS OF THE

 M = Molecular weight. ρ_{20} = Density at 20°C, in g./ml. α = Coefficient of cubical expansion, per Centigrade degree.

m.p. = Melting-point, degrees Centigrade.

c.p. = Crystallizing-point, degrees Centigrade.

b.p. = Boiling-point at 760 mm, degrees Centigrade.

C.V. = Gross calorific value, in g cal./g.

No.	Constituent	Formula	M	ρ_{20}	α
1	Anthracene	$C_{14}H_{10}$	178.22	1.250	0.000801
2	Benzene	C_6H_6	78.11	0.879	0.00124
3	Carbazole	$C_{12}H_9N$	167.20		
4	Carbon disulphide ..	CS_2	76.13	1.263	0.00120
5	Catechol; pyrocatechol	$C_6H_4(OH)_2$	110.11	1.344	
6	Coumarone	C_8H_6O	118.13	1.091	
7	<i>o</i> -Cresol	$CH_3C_6H_4OH$	108.13	1.051	
8	<i>m</i> -Cresol	$CH_3C_6H_4OH$	108.13	1.035	
9	<i>p</i> -Cresol	$CH_3C_6H_4OH$	108.13	1.035	
10	Fluorene	$C_{13}H_{10}$	166.21		
11	Indene	C_9H_8	116.15	1.006	
12	Naphthalene	$C_{10}H_8$	128.16	1.14	0.000853
13	α -Naphthol	$C_{10}H_7OH$	144.16	1.285	
14	β -Naphthol	$C_{10}H_7OH$	144.16	1.272	
15	Phenanthrene	$C_{14}H_{10}$	178.22	1.150	
16	Phenol	C_6H_5OH	94.11	1.073	0.00109
17	α -Picoline	$CH_3C_5H_4N$	93.12	0.945	
18	β -Picoline	$CH_3C_5H_4N$	93.12	0.957	
19	γ -Picoline	$CH_3C_5H_4N$	93.12	0.953	
20	Pyridine	C_5H_5N	79.10	0.9828	0.00097
21	Quinol; hydroquinone	$C_6H_4(OH)_2$	110.11	1.358	
22	Resorcinol	$C_6H_4(OH)_2$	110.05	1.28	
23	Thiophen	C_4H_4S	84.13	1.065	0.00112

MORE COMMON CONSTITUENTS OF COAL TAR

- c = Specific heat, in g cal./g.
 = Latent heat of fusion, in g cal./g.
 = Latent heat of vaporization at b.p., in g cal./g.

NOTE: The following data are recommended from among those at the Committee's disposal in published and previously unpublished records. The Committee will be grateful if any new data are brought to its notice.

No.	m.p./c.p.	b.p.	C.V.	c	l_f	l_v
1	218	340	9510	0.36/120°	38.7	
2	5.5	80.1	10030	0.406/20°	30.1	94.3
3	247	351.5	8820		42.1	
4	-111.6	46.3	3240	0.240/20°		84
5	105	245		0.278	49.4	
6		175				
7	31.0	191.0	8146	0.497/0°-120°		
8	12.0	202.2	8157	0.551/21°-197°		100.7
9	34.7	202.0	8152		26.3	
10	116	295	9540			
11	-2	182.4				
12	80.22	218	9614	0.313/0°	35.6	75.5
13	94	279		0.388/0°		
14	122	286		0.403/0°		
15	101	332	9506	0.30/solid	24.2	
16	41.0	181.7	7787	0.562/14°-26°	29.0	
17	-70	128-129	8762	0.42/76°		90.8
18		143.8	8730	0.42/76°		96.7
19		143.1	8768			
20	-42	115.3	8349	0.40/liquid	24.99	101.4
21	170.5	286.2				
22	110	278.4	6210	0.269	46.2	
23	-40	85	7975		14.1	

TABLE 42—PHYSICAL CONSTANTS OF THE

No.	Constituent	Formula	<i>M</i>	ρ_{20}	α
24	α -Thiotolene	$\text{CH}_3\text{C}_4\text{H}_3\text{S}$	98.16	1.016	
25	β -Thiotolene	$\text{CH}_3\text{C}_4\text{H}_3\text{S}$	98.16	1.025	
26	Toluene	$\text{C}_6\text{H}_5\text{CH}_3$	92.13	0.866	0.00110
27	<i>o</i> -Xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	0.880	0.00102
28	<i>m</i> -Xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	0.864	
29	<i>p</i> -Xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	0.861	
30	<i>o</i> -3-Xylenol (1 : 2-dimethyl-3-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16		
31	<i>o</i> -4-Xylenol (1 : 2-dimethyl-4-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16		
32	<i>m</i> -2-Xylenol (1 : 3-dimethyl-2-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16		
33	<i>m</i> -4-Xylenol (1 : 3-dimethyl-4-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16	1.026	
34	<i>m</i> -5-Xylenol (1 : 3-dimethyl-5-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16	1.016	
35	<i>p</i> -2-Xylenol (1 : 4-dimethyl-2-hydroxybenzene)	$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{OH}$	122.16	1.026	
36					
37					
38					
39					
40					
41					
42					
43					
44					

MORE COMMON CONSTITUENTS OF COAL TAR (*continued*)

No.	m.p./c.p.	b.p.	C.V.	c	l_f	l_v
24	-51	112				
25		115				
26	-95.0	110.5	10160	0.399/12°-99°	17.2	86.5
27	-25.7	144	10270	0.409/20°	29.3	83
28	-50	139.2	10270	0.394/20°	25.8	82
29	13.2	138.4	10270	0.398/20°	38.1	81
30	75.0	218.0				
31	65.0	227.0	8480			
32	45.0	201.0				
33	26.0	211.5	8500			
34	64.0	221.5				
35	75.0	211.5	8480			
36						
37						
38						
39						
40						
41						
42						
43						
44						

DENSITIES OF AQUEOUS SOLUTIONS OF SULPHURIC ACID AND SODIUM HYDROXIDE

Attention is drawn to the extensive tables given in B.S. 753—1937: Density-Composition Tables for Aqueous Solutions of Sulphuric Acid, and a similar British Standard (which it is expected will be published towards the end of 1938) entitled: Density-Composition Tables for Aqueous Solutions of Caustic Soda.

Other tables which might be of interest to users of "Standard Methods" are those (in course of preparation) for nitric acid and hydrochloric acid.

DEFINITION OF AND RELATION BETWEEN RECOGNIZED HEAT UNITS

One Mean British Thermal Unit is $\frac{1}{180}$ th part of the heat required to raise the temperature of one pound weight of water from 32° to 212°F.

One Mean Gram Calorie is $\frac{1}{100}$ th part of the heat required to raise the temperature of one gram of water from 0° to 100°C; (the 15° calorie differs from the mean gram calorie only by 1 in 4,000, which is too small to require any correction for all practical purposes).

One Mean Pound Calorie is $\frac{1}{100}$ th part of the heat required to raise the temperature of one pound weight of water from 0° to 100°C.

One Mean British Thermal Unit	251.996 Mean Gram Calories.
One Mean Pound Calorie	1.8 British Thermal Units
and	453.592 Mean Gram Calories.

RELATION BETWEEN ATMOSPHERIC PRESSURE AND ALTITUDE

If it be assumed that at mean sea level the air temperature is 15°C and the atmospheric pressure 760 mm, the pressure P_h mm at any height h metres above sea level not greater than 11,000 metres is given approximately by:—

$$\frac{P_h}{760} = \left\{ \frac{288 - 0.0065h}{288} \right\}^{5.256}$$

CALCULATION OF VOLUME OF CONTENTS OF HORIZONTAL CYLINDRICAL CONTAINERS WITH OR WITHOUT DISHED ENDS

(After Cross, "Handbook of Petroleum, Asphalt and Natural Gas," 1928, p. 169.)

If

- l = the length of the tank in inches;
- d = the diameter of the tank and the radius of curvature of the ends in inches;
- f = the liquid depth of the contents of the tank in inches;
- c = the total capacity of the cylindrical portion of the tank in imperial gallons; and
- b = the total capacity of both dished ends in imperial gallons,

then

- c = $0.002832d^3l$ gallons;
- b = $0.0003885d^3$ gallons; and

$$\frac{100f}{d} = \text{the liquid depth as a percentage (\%d) of the total diameter.}$$

The volume of the liquid contents of a tank may be calculated, using Tables 43 and 44, pages 386 to 389, as shewn in the following example:—

If, in the case of a particular tank,

- l = 360 inches;
- d = 108 inches; and
- f = 40 inches;

then

$$\begin{aligned} c &= 11,890 \text{ gallons;} \\ b &= 489 \text{ gallons;} \\ c + b &= 12,379 \text{ gallons, the total capacity of the tank; and} \\ \%d &= \frac{100f}{d} = 37.0. \end{aligned}$$

From Table 43, at $\%d = 37.0$, $\%c = 33.64$ and therefore the volume of the liquid in the cylindrical portion of the tank is 33.64% of 11,890 or .. 3,999 gal.

From Table 44, at $\%d = 37.0$, $\%b = 28.90$ and therefore the volume of the liquid in the dished ends of the tank is 28.90% of 489, or 141.4 gal.

The total volume of the contents is therefore .. 4,140.4 gal.

CALCULATION OF VOLUME OF CONTENTS OF HORIZONTAL CYLINDRICAL CONTAINERS WITH HEMISPHERICAL ENDS

Considerations closely parallel to the foregoing apply in the case of a tank with hemispherical ends, but d is in that case the diameter of the tank and *twice* the radius of curvature of the ends in inches; b, the total capacity of the ends, is then $0.001887d^3$; and Table 45, page 390 is used instead of Table 44.

TABLE 43: GAUGING OF HORIZONTAL

% d = liquid depth as percentage of total diameter of tank.

% d	% c	% d	% c	% d	% c	% d	% c	% d	% c
0.1	0.0053	5.1	1.9250	10.1	5.2805	15.1	9.497	20.1	14.341
0.2	0.0152	5.2	1.9814	10.2	5.3580	15.2	9.588	20.2	14.444
0.3	0.0279	5.3	2.0383	10.3	5.4350	15.3	9.679	20.3	14.547
0.4	0.0429	5.4	2.0956	10.4	5.5122	15.4	9.771	20.4	14.649
0.5	0.0600	5.5	2.1535	10.5	5.5902	15.5	9.863	20.5	14.751
0.6	0.0788	5.6	2.2116	10.6	5.6690	15.6	9.956	20.6	14.854
0.7	0.0992	5.7	2.2705	10.7	5.7472	15.7	10.048	20.7	14.957
0.8	0.1212	5.8	2.3297	10.8	5.8258	15.8	10.142	20.8	15.060
0.9	0.1445	5.9	2.3895	10.9	5.9050	15.9	10.234	20.9	15.163
1.0	0.1692	6.0	2.4497	11.0	5.9848	16.0	10.327	21.0	15.267
1.1	0.1952	6.1	2.5105	11.1	6.0645	16.1	10.422	21.1	15.371
1.2	0.2223	6.2	2.5715	11.2	6.1445	16.2	10.515	21.2	15.475
1.3	0.2508	6.3	2.6333	11.3	6.2255	16.3	10.609	21.3	15.579
1.4	0.2800	6.4	2.6952	11.4	6.3060	16.4	10.703	21.4	15.683
1.5	0.3104	6.5	2.7579	11.5	6.3870	16.5	10.797	21.5	15.787
1.6	0.3419	6.6	2.8211	11.6	6.4685	16.6	10.893	21.6	15.892
1.7	0.3744	6.7	2.8845	11.7	6.5500	16.7	10.988	21.7	15.998
1.8	0.4077	6.8	2.9483	11.8	6.6320	16.8	11.082	21.8	16.101
1.9	0.4421	6.9	3.0127	11.9	6.7145	16.9	11.178	21.9	16.206
2.0	0.4773	7.0	3.0771	12.0	6.7970	17.0	11.273	22.0	16.312
2.1	0.5134	7.1	3.1426	12.1	6.8793	17.1	11.369	22.1	16.418
2.2	0.5501	7.2	3.2082	12.2	6.9630	17.2	11.465	22.2	16.524
2.3	0.5881	7.3	3.2742	12.3	7.0460	17.3	11.561	22.3	16.630
2.4	0.6263	7.4	3.3408	12.4	7.1305	17.4	11.657	22.4	16.737
2.5	0.6660	7.5	3.4075	12.5	7.2145	17.5	11.754	22.5	16.842
2.6	0.7061	7.6	3.4749	12.6	7.2990	17.6	11.851	22.6	16.949
2.7	0.7470	7.7	3.5426	12.7	7.3830	17.7	11.949	22.7	17.055
2.8	0.7886	7.8	3.6106	12.8	7.4680	17.8	12.046	22.8	17.161
2.9	0.8310	7.9	3.6790	12.9	7.5540	17.9	12.143	22.9	17.269
3.0	0.8742	8.0	3.7480	13.0	7.6390	18.0	12.240	23.0	17.376
3.1	0.9179	8.1	3.8171	13.1	7.7245	18.1	12.338	23.1	17.483
3.2	0.9625	8.2	3.8869	13.2	7.8110	18.2	12.437	23.2	17.590
3.3	1.0075	8.3	3.9570	13.3	7.8970	18.3	12.535	23.3	17.698
3.4	1.0533	8.4	4.0276	13.4	7.9840	18.4	12.633	23.4	17.806
3.5	1.0998	8.5	4.0983	13.5	8.0710	18.5	12.732	23.5	17.913
3.6	1.1470	8.6	4.1696	13.6	8.1580	18.6	12.831	23.6	18.022
3.7	1.1947	8.7	4.2411	13.7	8.2450	18.7	12.930	23.7	18.130
3.8	1.2432	8.8	4.3131	13.8	8.3330	18.8	13.030	23.8	18.240
3.9	1.2921	8.9	4.3855	13.9	8.4210	18.9	13.130	23.9	18.348
4.0	1.3418	9.0	4.4582	14.0	8.5090	19.0	13.229	24.0	18.457
4.1	1.3920	9.1	4.5312	14.1	8.5975	19.1	13.329	24.1	18.566
4.2	1.4429	9.2	4.6045	14.2	8.6860	19.2	13.429	24.2	18.675
4.3	1.4941	9.3	4.6782	14.3	8.7755	19.3	13.529	24.3	18.784
4.4	1.5461	9.4	4.7525	14.4	8.8645	19.4	13.630	24.4	18.892
4.5	1.5986	9.5	4.8270	14.5	8.9545	19.5	13.731	24.5	19.010
4.6	1.6515	9.6	4.9015	14.6	9.0440	19.6	13.832	24.6	19.110
4.7	1.7052	9.7	4.9769	14.7	9.1345	19.7	13.934	24.7	19.220
4.8	1.7594	9.8	5.0523	14.8	9.2240	19.8	14.035	24.8	19.330
4.9	1.8142	9.9	5.1280	14.9	9.3150	19.9	14.136	24.9	19.440
5.0	1.8693	10.0	5.2040	15.0	9.406	20.0	14.238	25.0	19.551

PHYSICAL DATA

387

CYLINDRICAL TANKS

% c = volume of contents of cylindrical portion as percentage of total capacity of cylindrical portion.

% d	% c	% d	% c	% d	% c	% d	% c	% d	% c
25.1	19.662	30.1	25.350	35.1	31.314	40.1	37.480	45.1	43.775
25.2	19.773	30.2	25.467	35.2	31.436	40.2	37.606	45.2	43.902
25.3	19.884	30.3	25.584	35.3	31.558	40.3	37.731	45.3	44.028
25.4	19.995	30.4	25.701	35.4	31.680	40.4	37.856	45.4	44.155
25.5	20.106	30.5	25.818	35.5	31.802	40.5	37.981	45.5	44.282
25.6	20.217	30.6	25.935	35.6	31.924	40.6	38.106	45.6	44.409
25.7	20.328	30.7	26.052	35.7	32.046	40.7	38.231	45.7	44.538
25.8	20.439	30.8	26.170	35.8	32.168	40.8	38.355	45.8	44.663
25.9	20.550	30.9	26.288	35.9	32.290	40.9	38.479	45.9	44.790
26.0	20.661	31.0	26.407	36.0	32.412	41.0	38.604	46.0	44.918
26.1	20.773	31.1	26.524	36.1	32.534	41.1	38.730	46.1	45.043
26.2	20.886	31.2	26.642	36.2	32.657	41.2	38.856	46.2	45.171
26.3	20.998	31.3	26.760	36.3	32.780	41.3	38.982	46.3	45.298
26.4	21.110	31.4	26.878	36.4	32.902	41.4	39.108	46.4	45.424
26.5	21.222	31.5	26.996	36.5	33.025	41.5	39.233	46.5	45.550
26.6	21.334	31.6	27.114	36.6	33.147	41.6	39.358	46.6	45.678
26.7	21.447	31.7	27.232	36.7	33.269	41.7	39.482	46.7	45.803
26.8	21.560	31.8	27.351	36.8	33.392	41.8	39.608	46.8	45.930
26.9	21.672	31.9	27.470	36.9	33.515	41.9	39.735	46.9	46.058
27.0	21.785	32.0	27.589	37.0	33.638	42.0	39.862	47.0	46.183
27.1	21.898	32.1	27.708	37.1	33.762	42.1	39.988	47.1	46.311
27.2	22.011	32.2	27.827	37.2	33.885	42.2	40.114	47.2	46.438
27.3	22.125	32.3	27.946	37.3	34.008	42.3	40.240	47.3	46.565
27.4	22.239	32.4	28.065	37.4	34.131	42.4	40.365	47.4	46.693
27.5	22.353	32.5	28.184	37.5	34.254	42.5	40.490	47.5	46.819
27.6	22.467	32.6	28.302	37.6	34.377	42.6	40.615	47.6	46.947
27.7	22.581	32.7	28.422	37.7	34.501	42.7	40.741	47.7	47.074
27.8	22.695	32.8	28.543	37.8	34.625	42.8	40.869	47.8	47.201
27.9	22.810	32.9	28.660	37.9	34.749	42.9	40.994	47.9	47.329
28.0	22.923	33.0	28.781	38.0	34.873	43.0	41.120	48.0	47.457
28.1	23.038	33.1	28.899	38.1	34.996	43.1	41.246	48.1	47.583
28.2	23.152	33.2	29.020	38.2	35.119	43.2	41.372	48.2	47.710
28.3	23.266	33.3	29.140	38.3	35.242	43.3	41.499	48.3	47.837
28.4	23.380	33.4	29.260	38.4	35.368	43.4	41.628	48.4	47.965
28.5	23.494	33.5	29.380	38.5	35.491	43.5	41.749	48.5	48.093
28.6	23.611	33.6	29.500	38.6	35.615	43.6	41.876	48.6	48.220
28.7	23.728	33.7	29.620	38.7	35.739	43.7	42.002	48.7	48.348
28.8	23.842	33.8	29.740	38.8	35.865	43.8	42.129	48.8	48.475
28.9	23.957	33.9	29.860	38.9	35.988	43.9	42.257	48.9	48.603
29.0	24.072	34.0	29.981	39.0	36.110	44.0	42.383	49.0	48.729
29.1	24.187	34.1	30.102	39.1	36.234	44.1	42.510	49.1	48.857
29.2	24.302	34.2	30.223	39.2	36.359	44.2	42.637	49.2	48.983
29.3	24.418	34.3	30.344	39.3	36.483	44.3	42.762	49.3	49.112
29.4	24.535	34.4	30.465	39.4	36.608	44.4	42.890	49.4	49.239
29.5	24.651	34.5	30.587	39.5	36.732	44.5	43.018	49.5	49.366
29.6	24.769	34.6	30.708	39.6	36.856	44.6	43.142	49.6	49.494
29.7	24.884	34.7	30.829	39.7	36.981	44.7	43.268	49.7	49.621
29.8	25.000	34.8	30.950	39.8	37.106	44.8	43.397	49.8	49.748
29.9	25.116	34.9	31.071	39.9	37.230	44.9	43.521	49.9	49.877
30.0	25.233	35.0	31.192	40.0	37.355	45.0	43.648	50.0	50.000

TABLE 44: GAUGING OF DISHEED ENDS

% d = liquid depth as percentage of total diameter of tank.

% d	% b	% d	% b	% d	% b	% d	% b	% d	% b
0.1	0.00	5.1	0.32	10.1	1.62	15.1	4.18	20.1	7.99
0.2	0.00	5.2	0.34	10.2	1.66	15.2	4.24	20.2	8.09
0.3	0.00	5.3	0.36	10.3	1.69	15.3	4.31	20.3	8.19
0.4	0.00	5.4	0.38	10.4	1.73	15.4	4.38	20.4	8.28
0.5	0.01	5.5	0.40	10.5	1.77	15.5	4.44	20.5	8.38
0.6	0.01	5.6	0.41	10.6	1.81	15.6	4.50	20.6	8.46
0.7	0.01	5.7	0.43	10.7	1.85	15.7	4.57	20.7	8.54
0.8	0.01	5.8	0.45	10.8	1.89	15.8	4.63	20.8	8.63
0.9	0.01	5.9	0.47	10.9	1.94	15.9	4.70	20.9	8.72
1.0	0.01	6.0	0.49	11.0	1.98	16.0	4.77	21.0	8.81
1.1	0.01	6.1	0.50	11.1	2.03	16.1	4.83	21.1	8.89
1.2	0.01	6.2	0.52	11.2	2.07	16.2	4.90	21.2	8.97
1.3	0.01	6.3	0.53	11.3	2.11	16.3	4.96	21.3	9.06
1.4	0.02	6.4	0.54	11.4	2.15	16.4	5.03	21.4	9.15
1.5	0.02	6.5	0.56	11.5	2.20	16.5	5.10	21.5	9.24
1.6	0.02	6.6	0.58	11.6	2.24	16.6	5.17	21.6	9.34
1.7	0.02	6.7	0.60	11.7	2.29	16.7	5.25	21.7	9.44
1.8	0.02	6.8	0.62	11.8	2.33	16.8	5.32	21.8	9.54
1.9	0.02	6.9	0.64	11.9	2.38	16.9	5.40	21.9	9.64
2.0	0.02	7.0	0.66	12.0	2.43	17.0	5.48	22.0	9.74
2.1	0.03	7.1	0.68	12.1	2.48	17.1	5.55	22.1	9.84
2.2	0.03	7.2	0.70	12.2	2.54	17.2	5.63	22.2	9.93
2.3	0.04	7.3	0.73	12.3	2.59	17.3	5.71	22.3	10.03
2.4	0.04	7.4	0.75	12.4	2.65	17.4	5.78	22.4	10.12
2.5	0.05	7.5	0.78	12.5	2.70	17.5	5.86	22.5	10.22
2.6	0.05	7.6	0.81	12.6	2.75	17.6	5.94	22.6	10.32
2.7	0.06	7.7	0.84	12.7	2.80	17.7	6.02	22.7	10.42
2.8	0.06	7.8	0.87	12.8	2.85	17.8	6.10	22.8	10.52
2.9	0.07	7.9	0.90	12.9	2.90	17.9	6.17	22.9	10.62
3.0	0.07	8.0	0.92	13.0	2.95	18.0	6.25	23.0	10.72
3.1	0.08	8.1	0.95	13.1	3.01	18.1	6.33	23.1	10.82
3.2	0.08	8.2	0.98	13.2	3.06	18.2	6.41	23.2	10.93
3.3	0.09	8.3	1.01	13.3	3.12	18.3	6.49	23.3	11.04
3.4	0.10	8.4	1.05	13.4	3.17	18.4	6.57	23.4	11.14
3.5	0.11	8.5	1.08	13.5	3.22	18.5	6.64	23.5	11.25
3.6	0.12	8.6	1.11	13.6	3.28	18.6	6.72	23.6	11.36
3.7	0.13	8.7	1.14	13.7	3.33	18.7	6.80	23.7	11.47
3.8	0.14	8.8	1.17	13.8	3.39	18.8	6.88	23.8	11.58
3.9	0.15	8.9	1.20	13.9	3.44	18.9	6.96	23.9	11.69
4.0	0.16	9.0	1.23	14.0	3.50	19.0	7.05	24.0	11.80
4.1	0.17	9.1	1.26	14.1	3.56	19.1	7.13	24.1	11.90
4.2	0.18	9.2	1.30	14.2	3.62	19.2	7.21	24.2	12.01
4.3	0.19	9.3	1.33	14.3	3.68	19.3	7.29	24.3	12.12
4.4	0.20	9.4	1.36	14.4	3.74	19.4	7.37	24.4	12.22
4.5	0.21	9.5	1.40	14.5	3.80	19.5	7.46	24.5	12.32
4.6	0.22	9.6	1.43	14.6	3.87	19.6	7.55	24.6	12.43
4.7	0.24	9.7	1.46	14.7	3.93	19.7	7.63	24.7	12.54
4.8	0.26	9.8	1.50	14.8	4.00	19.8	7.72	24.8	12.66
4.9	0.28	9.9	1.54	14.9	4.06	19.9	7.81	24.9	12.77
5.0	0.30	10.0	1.58	15.0	4.12	20.0	7.90	25.0	12.89

PHYSICAL DATA

389

OF HORIZONTAL CYLINDRICAL TANKS

% b = total volume of contents of both dished ends as percentage of total capacity of both dished ends.

% d	% b	% d	% b	% d	% b	% d	% b	% d	% b
25.1	12.95	30.1	19.06	35.1	26.05	40.1	33.74	45.1	41.77
25.2	13.06	30.2	19.19	35.2	26.20	40.2	33.90	45.2	41.94
25.3	13.17	30.3	19.32	35.3	26.35	40.3	34.05	45.3	42.11
25.4	13.29	30.4	19.43	35.4	26.50	40.4	34.20	45.4	42.28
25.5	13.40	30.5	19.55	35.5	26.65	40.5	34.35	45.5	42.45
25.6	13.51	30.6	19.68	35.6	26.80	40.6	34.50	45.6	42.61
25.7	13.63	30.7	19.81	35.7	26.95	40.7	34.65	45.7	42.77
25.8	13.75	30.8	19.94	35.8	27.10	40.8	34.80	45.8	42.93
25.9	13.87	30.9	20.07	35.9	27.25	40.9	34.95	45.9	43.09
26.0	13.98	31.0	20.22	36.0	27.40	41.0	35.10	46.0	43.25
26.1	14.10	31.1	20.37	36.1	27.55	41.1	35.26	46.1	43.41
26.2	14.22	31.2	20.52	36.2	27.70	41.2	35.42	46.2	43.57
26.3	14.34	31.3	20.67	36.3	27.84	41.3	35.58	46.3	43.73
26.4	14.46	31.4	20.82	36.4	27.99	41.4	35.75	46.4	43.89
26.5	14.58	31.5	20.97	36.5	28.13	41.5	35.92	46.5	44.05
26.6	14.70	31.6	21.11	36.6	28.28	41.6	36.08	46.6	44.22
26.7	14.82	31.7	21.35	36.7	28.43	41.7	36.24	46.7	44.38
26.8	14.94	31.8	21.39	36.8	28.59	41.8	36.39	46.8	44.54
26.9	15.06	31.9	21.52	36.9	28.75	41.9	36.55	46.9	44.71
27.0	15.19	32.0	21.65	37.0	28.90	42.0	36.70	47.0	44.88
27.1	15.31	32.1	21.79	37.1	29.05	42.1	36.86	47.1	45.05
27.2	15.43	32.2	21.93	37.2	29.20	42.2	37.02	47.2	45.23
27.3	15.56	32.3	22.07	37.3	29.35	42.3	37.18	47.3	45.31
27.4	15.68	32.4	22.20	37.4	29.50	42.4	37.34	47.4	45.59
27.5	15.80	32.5	22.34	37.5	29.65	42.5	37.50	47.5	45.77
27.6	15.92	32.6	22.47	37.6	29.80	42.6	37.67	47.6	45.95
27.7	16.04	32.7	22.60	37.7	29.95	42.7	37.83	47.7	46.12
27.8	16.16	32.8	22.74	37.8	30.10	42.8	37.99	47.8	46.29
27.9	16.28	32.9	22.87	37.9	30.26	42.9	38.16	47.9	46.46
28.0	16.40	33.0	23.00	38.0	30.42	43.0	38.32	48.0	46.63
28.1	16.53	33.1	23.14	38.1	30.58	43.1	38.49	48.1	46.80
28.2	16.65	33.2	23.28	38.2	30.74	43.2	38.65	48.2	46.96
28.3	16.77	33.3	23.41	38.3	30.91	43.3	38.81	48.3	47.13
28.4	16.90	33.4	23.55	38.4	31.08	43.4	38.97	48.4	47.30
28.5	17.02	33.5	23.69	38.5	31.25	43.5	39.13	48.5	47.46
28.6	17.14	33.6	23.84	38.6	31.40	43.6	39.30	48.6	47.62
28.7	17.27	33.7	23.99	38.7	31.56	43.7	39.46	48.7	47.77
28.8	17.39	33.8	24.15	38.8	31.72	43.8	39.62	48.8	47.93
28.9	17.51	33.9	24.31	38.9	31.87	43.9	39.78	48.9	48.09
29.0	17.63	34.0	24.45	39.0	32.02	44.0	39.95	49.0	48.25
29.1	17.76	34.1	24.59	39.1	32.16	44.1	40.12	49.1	48.42
29.2	17.89	34.2	24.74	39.2	32.31	44.2	40.29	49.2	48.59
29.3	18.02	34.3	24.89	39.3	32.46	44.3	40.46	49.3	48.76
29.4	18.15	34.4	25.05	39.4	32.60	44.4	40.62	49.4	48.93
29.5	18.27	34.5	25.20	39.5	32.75	44.5	40.79	49.5	49.10
29.6	18.40	34.6	25.36	39.6	32.91	44.6	40.95	49.6	49.28
29.7	18.53	34.7	25.52	39.7	33.06	44.7	41.11	49.7	49.46
29.8	18.66	34.8	25.68	39.8	33.32	44.8	41.27	49.8	49.64
29.9	18.80	34.9	25.84	39.9	33.45	44.9	41.44	49.9	49.82
30.0	18.93	35.0	25.90	40.0	33.58	45.0	41.60	50.0	50.00

TABLE 45: GAUGING OF HEMISPHERICAL ENDS OF HORIZONTAL CYLINDRICAL TANKS

% d = liquid depth as percentage of total diameter of tank.

% b = total volume of contents of both hemispherical ends as percentage of total capacity of both hemispherical ends.

% d	0	10	20	30	40	50	60	70	80	90
	% b	% b	% b	% b	% b	% b	% b	% b	% b	% b
0	0.000	2.80	10.40	21.60	35.20	50.00	64.80	78.40	89.60	97.20
0.5	0.007	3.08	10.89	22.23	35.92	50.75	65.52	79.03	90.07	97.46
1.0	0.030	3.36	11.38	22.87	36.65	51.50	66.23	79.65	90.54	97.72
1.5	0.067	3.66	11.88	23.52	37.37	52.25	66.94	80.26	91.00	97.96
2.0	0.120	3.97	12.39	24.17	38.10	53.00	67.65	80.87	91.45	98.18
2.5	0.18	4.30	12.91	24.82	38.83	53.75	68.36	81.47	91.88	98.40
3.0	0.26	4.63	13.44	25.48	39.57	54.50	69.06	82.07	92.31	98.60
3.5	0.36	4.98	13.98	26.15	40.30	55.24	69.76	82.65	92.73	98.79
4.0	0.47	5.33	14.52	26.82	41.04	55.99	70.45	83.23	93.14	98.96
4.5	0.59	5.70	15.07	27.49	41.78	56.74	71.14	83.81	93.54	99.13
5.0	0.72	6.07	15.62	28.17	42.52	57.48	71.83	84.38	93.93	99.28
5.5	0.87	6.46	16.19	28.86	43.26	58.22	72.51	84.93	94.30	99.41
6.0	1.04	6.86	16.77	29.55	44.01	58.96	73.18	85.48	94.67	99.53
6.5	1.21	7.27	17.35	30.24	44.76	59.70	73.85	86.02	95.02	99.64
7.0	1.40	7.69	17.93	30.94	45.50	60.43	74.52	86.56	95.37	99.74
7.5	1.60	8.12	18.53	31.64	46.25	61.17	75.18	87.09	95.70	99.82
8.0	1.82	8.55	19.13	32.35	47.00	61.90	75.83	87.61	96.03	99.88
8.5	2.04	9.00	19.74	33.06	47.75	62.63	76.48	88.12	96.34	99.93
9.0	2.28	9.46	20.35	33.77	48.50	63.35	77.13	88.62	96.64	99.97
9.5	2.54	9.93	20.97	34.48	49.25	64.08	77.77	89.11	96.92	99.99

NOTE: Interpolation should suffice for all practical purposes when intermediate points are required, but the following relationship may occasionally be found useful:—

$$\% b = (300\% d)^3 - 2(\% d)^3 / 10,000$$

MEASUREMENT OF LIQUIDS IN BULK

1. Measurements Based on Density Determinations

The density of a liquid provides a simple basis for computing the volume of a known weight, or the weight of a known volume. Tables for facilitating such computations appear below and the conversion data, being based directly upon density, are applicable to densities however determined, e.g. whether by means of the standard density hydrometers or with a density bottle.

(a) Imperial System:

Table 46, below, provides a convenient means of converting density in g/ml to lb weight in air per imperial gallon. To obtain lb per imperial gallon the correction in the second column of Table 46 is added to the density in the first column and the result multiplied by ten is the weight in air in lb of the quantity of the liquid which occupies one imperial gallon at $t^{\circ}\text{C}$. For densities intermediate between those tabulated, inspection serves to indicate the appropriate correction.

TABLE 46: CONVERSION OF THE DENSITY OF A LIQUID IN G/ML TO LB WEIGHT IN AIR PER IMPERIAL GALLON

<i>Density in g/ml at $t^{\circ}\text{C}$</i>	<i>Correction (c) for the weight in air in lb of the quantity of liquid occupying one imperial gallon at $t^{\circ}\text{C}$</i>
0.60	2
0.65	3
0.70	4
0.75	6
0.80	7
0.85	8
0.90	9
0.95	10
1.00	12
1.05	13
1.10	14
1.15	15
1.20	16
1.25	17
1.30	19

NOTE: The corrections are expressed as units in the fourth decimal place and are positive in sign.

Examples

(i) WEIGHT OF A MEASURED VOLUME OF LIQUID:

Observations:

Temperature of sample of liquid ($t^{\circ}\text{C}$)	..	15 $^{\circ}\text{C}$
Density of sample of liquid at $t^{\circ}\text{C}$ (ρ_t)	..	0.7836 g/ml
Temperature of bulk of liquid ($t_1^{\circ}\text{C}$)	..	30 $^{\circ}\text{C}$
Volume of bulk of liquid at $t_1^{\circ}\text{C}$ (V_{t_1})	..	1023 gal.

Computations:

Density ρ_t	0.7836 g/ml
Correction (c) from Table 46	+0.0007

$$\rho_t + c = 0.7843$$

Weight (W_t) in air of a gallon of liquid at $t^{\circ}\text{C} = 10(\rho_t + c)$	7.843 lb
Coefficient of cubical expansion of liquid (α)* per Centigrade degree	0.00102
Volume (V_t) of bulk of liquid at $t^{\circ}\text{C}$, i.e. at 15 $^{\circ}\text{C}$, is $V_{t_1} [1 + \alpha (t - t_1)]$				
$= 1023 [1 + 0.00102 (15 - 30)]$				<u>1007.3 gal.</u>

Weight in air of bulk of liquid is

$$\begin{aligned} W_t \times V_t \\ = 7.843 \times 1007.3 \dots = 7900 \text{ lb} \end{aligned}$$

A great advantage is secured if it be arranged that the temperature at which the density of the sample is determined be the same as that of the bulk, for no knowledge of the coefficient of expansion of the liquid is then necessary and the computation is simplified as shown by the following example:—

Observations:

Temperature of sample of liquid ($t^{\circ}\text{C}$)	30 $^{\circ}\text{C}$
Density of sample of liquid at $t^{\circ}\text{C}$ (ρ_t)	0.7715 g/ml
Temperature of bulk of liquid ($t^{\circ}\text{C}$)	30 $^{\circ}\text{C}$
Volume of bulk of liquid at $t^{\circ}\text{C}$ (V_t)	1023 gal.

Computations:

Density ρ_t	0.7715 g/ml
Correction (c) from Table 46	+0.0007

$$\rho + c \dots = 0.7722$$

Weight (W_t) in air of a gallon of liquid at $t^{\circ}\text{C} = 10(\rho_t + c)$	7.722 lb
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Weight in air of bulk of liquid is

$$\begin{aligned} W_t \times V_t \\ = 7.722 \times 1023 \dots = 7900 \text{ lb} \end{aligned}$$

* A value of α typical of liquids of high coefficient of expansion has been used, but the value given is clearly not of general applicability. The value of α appropriate for the liquid must be used.

(ii) VOLUME OF A DETERMINED WEIGHT OF LIQUID:

It will be assumed that the volume which the bulk of the liquid would occupy at some standard reference temperature is required and 20°C has been used for this temperature.

Observations:

Temperature of sample of liquid ($t^{\circ}\text{C}$)	..	15°C
Density of sample of liquid at 15°C (ρ_t)	..	0.7755 g/ml
Weight of bulk of liquid (W)	7900 lb

Computations:

Density ρ_t	0.7755
Correction (c) from Table 46	+0.0007
		$\rho_t + c$	= 0.7762
Weight (W_t) in air of a gallon of liquid at $t^{\circ}\text{C} = 10(\rho_t + c)$	7.762 lb
Volume (V_t) of bulk of liquid at $t^{\circ}\text{C}$ is		W/W_t	
		= 7900/7.762	= 1017.8 gal.
Coefficient of cubical expansion of liquid (α) per Centigrade degree	0.00102
Volume (V_{20}) of bulk of liquid at 20°C is		$V_t [1 + \alpha (20 - t)]$	
		= 1017.8[1 + 0.00102 (20 - 15)]	= 1023 gal.

In obtaining the volume of a determined weight of liquid it is a great advantage if the temperature at which the density is determined be the same as the standard reference temperature at which the volume of the bulk of the liquid is required. No knowledge of the coefficient of cubical expansion of the liquid is then necessary and the computation is simplified as shown by the following example:—

Observations:

Temperature of sample of liquid ($t^{\circ}\text{C}$)	..	20°C
Density of sample of liquid at $t^{\circ}\text{C}$ (ρ_t)	..	0.7715 g/ml
Weight of bulk of liquid (W)	7900 lb

Computations:

Density ρ_t	0.7715
Correction (c) from Table 46	+0.0007
		$\rho_t + c$	= 0.7722
Weight (W_t) in air of a gallon of liquid at $t^{\circ}\text{C} = 10(\rho_t + c)$	7.722 lb
Volume (V_t) of bulk of liquid at $t^{\circ}\text{C}$, i.e. in this case at 20°C, is		W/W_t	
		= 7900/7.722	= 1023 gal.

(b) Metric System:

The density in g/ml of a liquid is the mass in grams of one millilitre of the liquid and is numerically equal to the mass in kilograms of one litre of the liquid and to within 3 parts in 100,000 of the mass in tonnes of 1 cubic metre of the liquid.

The corrections in Table 47, below, serve for conversion from a mass basis to a weight in air basis.

TABLE 47: CONVERSION OF DENSITY IN G/ML TO THE WEIGHT IN AIR IN GRAMS OF 1 ML OF THE LIQUID

<i>Density of liquid in g/ml at t°C</i>	<i>Correction (d) to give weight in air in grams of the quantity of liquid occupying 1 ml at t°C</i>
0.60 to 1.03	-11
1.04 to 1.72	-10

NOTE: The corrections are expressed as units in the fourth decimal place; being negative in sign they are to be subtracted from the determined density ρ_t .

By applying the appropriate correction from Table 47 to an observed density in g/ml at t°C, the weight in air in grams of the quantity of the liquid occupying 1 ml at t°C is obtained and this is numerically equal to the weight in air in kilograms of the quantity of the liquid occupying one litre at t°C and to within 3 parts in 100,000 to the weight in air in tonnes of the quantity of liquid occupying one cubic metre at t°C.

The computation of the weight of a known volume of liquid and of the volume of a known weight of liquid follow precisely similar lines to those given for imperial units. It is therefore unnecessary to repeat them in detail and the following example will suffice.

Observations:

Temperature of sample of liquid (t°C)	..	30°C
Density of sample of liquid at t°C (ρ_t)	..	0.7715 g/ml
Temperature of bulk of liquid (t°C)	..	30°C
Volume of bulk of liquid at t°C (V_t)	..	50.23 m ³

Computations:

Density ρ_t	0.7715 g/ml
Correction (d) from Table 47	-0.0011
$\rho_t + d$					<u>0.7704</u>

Weight (W_t) in air of a cubic metre of liquid
at t°C, i.e. in this case at 30°C

$$= (\rho_t + d) \dots \dots = 0.7704 \text{ tonnes}$$

Weight in air of bulk of liquid is

$$W_t \times V$$

$$= 0.7704 \times 50.23 \text{ tonnes} = 38.70 \text{ tonnes}$$

It will be seen that the above computation is precisely similar to that on page 392.

2. Measurements Based on Specific Gravity Determinations

The specific gravity of a liquid provides a simple basis for computing the volume of a known weight, or the weight of a known volume. Tables for facilitating such computations appear below and the conversion data, being based directly on the specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$, are applicable to such specific gravities however determined, e.g. whether by means of the S.T.P.T.C. specific gravity hydrometers or with a density bottle.

(a) Imperial System:

Table 48, below, provides a convenient means of converting specific gravities $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ to lb weight in air per imperial gallon. To obtain lb per imperial gallon the correction in the second column of Table 48 is applied to the specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ in the first column and the result multiplied by ten is the weight in air in lb of the quantity of the liquid which occupies one imperial gallon at $t^{\circ}\text{C}$. For specific gravities intermediate between those tabulated, inspection serves to indicate the appropriate correction.

TABLE 48: CONVERSION OF SPECIFIC GRAVITY $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ TO LB WEIGHT IN AIR PER IMPERIAL GALLON

<i>Specific gravity</i> $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$	<i>Correction (c_1) for the</i> <i>weight in air in lb of</i> <i>the quantity of liquid</i> <i>occupying one imperial</i> <i>gallon at $t^{\circ}\text{C}$</i>
0.60	-4
0.65	-3
0.70	-2
0.75	-1
0.80	-1
0.85	0
0.90	+1
0.95	+1
1.00	+2
1.05	+3
1.10	+3
1.15	+4
1.20	+5
1.25	+6
1.30	+6

NOTE: The corrections are expressed as units in the fourth place of decimals; when positive (+) they are to be added to and when negative (-) subtracted from the observed specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$.

Examples

(i) WEIGHT OF A MEASURED VOLUME OF LIQUID:

Observations:

Temperature of sample of liquid ($t^\circ\text{C}$)	..	20°C
Specific gravity of sample of liquid ($S_{t^\circ\text{C}/15.5^\circ\text{C}}$)	..	0.7803
Temperature of bulk of liquid ($t_1^\circ\text{C}$)	..	30°C
Volume of bulk of liquid at $t_1^\circ\text{C}$ (V_{t_1})	..	1023 gal.

Computations:

Specific gravity $S_{20^\circ\text{C}/15.5^\circ\text{C}}$	0.7803
Correction (c_1) from Table 48	-0.0001

$$S_{20^\circ\text{C}/15.5^\circ\text{C}} + c_1 = \underline{\underline{0.7802}}$$

$$\text{Weight (W}_t\text{) in air of a gallon of liquid at } t^\circ\text{C} = 10(S_{20^\circ\text{C}/15.5^\circ\text{C}} + c_1) = 7.802 \text{ lb}$$

$$\text{Coefficient of cubical expansion of liquid } (\alpha) * \text{ per Centigrade degree.} \dots = 0.00102$$

$$\text{Volume (V}_t\text{) of bulk of liquid at } t^\circ\text{C, i.e. at } 20^\circ\text{C, is}$$

$$V_{t_1}[1 + \alpha(t - t_1)] = 1023[1 + 0.00102(20 - 30)] = \underline{\underline{1012.6 \text{ gal.}}}$$

$$\text{Weight in air of bulk of liquid is}$$

$$W_t \times V_t = 7.802 \times 1012.6 = 7900 \text{ lb}$$

A great advantage is secured if it be arranged that the temperature at which the specific gravity $S_{t^\circ\text{C}/15.5^\circ\text{C}}$ of the sample is determined be the same as that of the bulk, for no knowledge of the coefficient of expansion of the liquid is then necessary and the computation is simplified as shown by the following example:—

Observations:

Temperature of sample of liquid ($t^\circ\text{C}$)	..	30°C
Specific gravity of sample of liquid ($S_{t^\circ\text{C}/15.5^\circ\text{C}}$)	..	0.7723
Temperature of bulk of liquid ($t^\circ\text{C}$)	..	30°C
Volume of bulk of liquid at $t^\circ\text{C}$ (V_t)	..	1023 gal.

Computations:

Specific gravity $S_{30^\circ\text{C}/15.5^\circ\text{C}}$	0.7723
Correction (c_1) from Table 48	-0.0001

$$S_{30^\circ\text{C}/15.5^\circ\text{C}} + c_1 = \underline{\underline{0.7722}}$$

$$\text{Weight (W}_t\text{) in air of a gallon of liquid at } t^\circ\text{C} = 10(S_{30^\circ\text{C}/15.5^\circ\text{C}} + c_1) = 7.722 \text{ lb}$$

$$\text{Weight in air of bulk of liquid is}$$

$$W_t \times V_t = 7.722 \times 1023 = \underline{\underline{7900 \text{ lb}}}$$

* A value of α typical of liquids of high coefficient of expansion has been used, but the value given is clearly not of general applicability. The value of α appropriate for the liquid must be used.

(ii) VOLUME OF A DETERMINED WEIGHT OF LIQUID:

It will be assumed that the volume which the bulk of the liquid would occupy at some standard reference temperature is required and 15.5°C has been used for this temperature.

Observations:

Temperature of sample of liquid (t°C) ..	20°C
Specific gravity of sample of liquid ($S_{t^{\circ}C/15.5^{\circ}C}$) ..	0.7689
Weight of bulk of liquid (W)	7900 lb

Computations:

Specific gravity $S_{20^{\circ}C/15.5^{\circ}C}$	0.7689
Correction (c_1) from Table 48	-0.0001
$S_{20^{\circ}C/15.5^{\circ}C} + c_1 =$	<u>0.7688</u>
Weight (W_t) in air of a gallon of liquid at t°C = $10(S_{t^{\circ}C/15.5^{\circ}C} + c_1)$	7.688 lb
Volume (V_t) of bulk of liquid at t°C, is W/W_t = $7900/7.688$	<u>1027.6 gal.</u>
Coefficient of cubical expansion of liquid (α) per Centigrade degree	0.00102
Volume ($V_{15.5}$) of bulk of liquid at 15.5°C is $V_t[1 + \alpha(15.5 - t)]$ = $1027.6[1 + 0.00102 \times (15.5 - 20)]$ =	<u>1023 gal.</u>

In obtaining the volume of a determined weight of liquid it is a great advantage if the temperature at which the specific gravity $S_{t^{\circ}C/15.5^{\circ}C}$ is determined be the same as the standard reference temperature at which the volume of the bulk of the liquid is required. No knowledge of the coefficient of cubical expansion of the liquid is then necessary and the computation is simplified as shewn by the following example:—

Observations:

Temperature of sample of liquid (t°C) ..	15.5°C
Specific gravity of sample of liquid ($S_{t^{\circ}C/15.5^{\circ}C}$) ..	0.7723
Weight of bulk of liquid (W)	7900 lb

Computations:

Specific gravity $S_{15.5^{\circ}C/15.5^{\circ}C}$	0.7723
Correction (c_1) from Table 48	-0.0001
$S_{15.5^{\circ}C/15.5^{\circ}C} + c_1 =$	0.7722
Weight (W_t) in air of a gallon of liquid at t°C = $10(S_{t^{\circ}C/15.5^{\circ}C} + c_1)$	7.722 lb
Volume (V_t) of bulk of liquid at t°C, i.e. in this case at 15.5°C, is W/W_t = $7900/7.722$	<u>1023 gal.</u>

(b) Metric System:

Table 49, below, shows the correction which when applied to the specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ of a liquid gives the weight in air in grams of the quantity of the liquid which occupies one millilitre at $t^{\circ}\text{C}$. The weight in grams of one millilitre is numerically equal to the weight in kilograms of 1 litre and to within 3 parts in 100,000 to the weight in tonnes of one cubic metre.

TABLE 49: CONVERSION OF SPECIFIC GRAVITY $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$ TO WEIGHT IN AIR IN GRAMS OF 1 ML

<i>Specific gravity $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$</i>	<i>Correction (d_1) to give weight in air in grams of the quantity of liquid occupying 1 ml at $t^{\circ}\text{C}$</i>
0.60	-17
0.65	-17
0.70	-18
0.75	-18
0.80	-18
0.85	-19
0.90	-19
0.95	-20
1.00	-20
1.05	-20
1.10	-21
1.15	-21
1.20	-22
1.25	-22
1.30	-22

NOTE: The corrections are expressed as units in the fourth place of decimals; being negative in sign they are subtracted from the determined specific gravities $S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$.

The computation of the weight of a known volume of liquid and of the volume of a known weight of liquid follow precisely similar lines to those given for imperial units. It is therefore unnecessary to repeat them in detail and the following example will suffice.

Observations:

Temperature of sample of liquid ($t^{\circ}\text{C}$)	..	30°C
Specific gravity of sample of liquid ($S_{t^{\circ}\text{C}/15.5^{\circ}\text{C}}$)	..	0.7722
Temperature of bulk of liquid ($t^{\circ}\text{C}$)	..	30°C
Volume of bulk of liquid at $t^{\circ}\text{C}$ (V_t)	..	50.23 m ³

Computations:

Specific gravity $S_{30^{\circ}\text{C}/15.5^{\circ}\text{C}}$	0.7722
Correction (d_1) from Table 49	-0.0018

$$S_{30^{\circ}\text{C}/15.5^{\circ}\text{C}} + d_1 = 0.7704$$

Weight (W_t) in air of a cubic metre of liquid
at $t^{\circ}\text{C}$, i.e. in this case at 30°C

$$= (S_{30^{\circ}\text{C}/15.5^{\circ}\text{C}} + d_1) \quad \dots = 0.7704 \text{ tonnes}$$

Weight in air of bulk of liquid is

$$W_t \times V_t$$

$$= 0.7704 \times 50.23 \text{ tonnes} = 38.70 \text{ tonnes}$$

It will be seen that the above computation is precisely similar to that on page 396.



TABLE 50: CONVERSION TABLE FOR
(With acknowledgments to

°F	°C	°F	°C	°F	°C	°F	°C	°F	°C
-45.9	-27.3	-5	-20.6	40	4.4	85	29.4	130	54.4
-50	-45.6	-4	-20.4	41	5	86	30	131	55
-49	-45	-3	-19.4	42	5.6	87	30.6	132	55.6
-48	-44.4	-2	-19	42.8	6	87.8	31	132.8	56
-47.2	-44	-1	-18.9	43	6.1	88	31.1	133	56.1
-47	-43.9	-1	-18.3	44	6.7	89	31.7	134	56.7
-46	-43.3	-0.4	-18	44.6	7	89.6	32	134.6	57
-45.4	-43	0	-17.8	45	7.2	90	32.2	135	57.2
-45	-42.8	1	-17.2	46	7.8	91	32.8	136	57.8
-44	-42.2	1.4	-17	46.4	8	91.4	33	136.4	58
-43.6	-42	2	-16.7	47	8.3	92	33.3	137	58.3
-43	-41.7	3	-16.1	48	8.9	93	33.9	138	58.9
-42	-41.1	3.6	-16	48.2	9	93.2	34	138.2	59
-41.8	-41	4	-15.6	49	9.4	94	34.4	139	59.4
-41	-40.6	5	-15	50	10	95	35	140	60
-40	-40	6	-14.4	51	10.6	96	35.6	141	60.6
-39	-39.4	6.8	-14	51.8	11	96.8	36	141.8	61
-38.2	-39	7	-13.9	52	11.1	97	36.1	142	61.1
-38	-38.9	8	-13.3	53	11.7	98	36.7	143	61.7
-37	-38.3	8.6	-13	53.6	12	98.6	37	143.6	62
-36.4	-38	9	-12.8	54	12.2	99	37.2	144	62.2
-36	-37.8	10	-12.2	55	12.8	100	37.8	145	62.8
-35	-37.2	10.4	-12	55.4	13	100.4	38	145.4	63
-34.6	-37	11	-11.7	56	13.3	101	38.3	146	63.3
-34	-36.7	12	-11.1	57	13.9	102	38.9	147	63.9
-33	-36.1	12.2	-11	57.2	14	102.2	39	147.2	64
-32.8	-36	13	-10.6	58	14.4	103	39.4	148	64.4
-32	-35.6	14	-10	59	15	104	40	149	65
-31	-35	15	-9.4	60	15.6	105	40.6	150	65.6
-30	-34.4	15.8	-9	60.8	16	105.8	41	150.8	66
-29.2	-34	16	-8.9	61	16.1	106	41.1	151	66.1
-29	-33.9	17	-8.3	62	16.7	107	41.7	152	66.7
-28	-33.3	17.6	-8	62.6	17	107.6	42	152.6	67
-27.4	-33	18	-7.8	63	17.2	108	42.2	153	67.2
-27	-32.8	19	-7.2	64	17.8	109	42.8	154	67.8
-26	-32.2	19.4	-7	64.4	18	109.4	43	154.4	68
-25.6	-32	20	-6.7	65	18.3	110	43.3	155	68.3
-25	-31.7	21	-6.1	66	18.9	111	43.9	156	68.9
-24	-31.1	21.2	-6	66.2	19	111.2	44	156.2	69
-23.8	-31	22	-5.6	67	19.4	112	44.4	157	69.4
-23	-30.6	23	-5	68	20	113	45	158	70
-22	-30	24	-4.4	69	20.6	114	45.6	159	70.6
-21	-29.4	24.8	-4	69.8	21	114.8	46	159.8	71
-20.2	-29	25	-3.9	70	21.1	115	46.1	160	71.1
-20	-28.9	26	-3.3	71	21.7	116	46.7	161	71.7
-19	-28.3	26.6	-3	71.6	22	116.6	47	161.6	72
-18.4	-28	27	-2.8	72	22.2	117	47.2	162	72.2
-18	-27.8	28	-2.3	73	22.8	118	47.8	163	72.8
-17	-27.2	28.4	-2	73.4	23	118.4	48	163.4	73
-16.6	-27	29	-1.7	74	23.3	119	48.3	164	73.3
-16	-26.7	30	-1	75	23.9	120	48.9	165	73.9
-15	-26.1	30.2	-1	75.2	24	120.2	49	165.2	74
-14.8	-26	31	-0.6	76	24.4	121	49.4	166	74.4
-14	-25.6	32	0	77	25	122	50	167	75
-13	-25	33	0.6	78	25.6	123	50.6	168	75.6
-12	-24.4	33.8	1	78.8	26	123.8	51	168.8	76
-11.2	-24	34	1.1	79	26.1	124	51.1	169	76.1
-11	-23.9	35	1.7	80	26.7	125	51.7	170	76.7
-10	-23.3	35.6	2	80.6	27	125.6	52	170.6	77
-9.4	-23	36	2.2	81	27.2	126	52.2	171	77.2
-9	-22.8	37	2.8	82	27.8	127	52.8	172	77.8
-8	-22.2	37.4	3	82.4	28	127.4	53	172.4	78
-7.6	-22	38	3.3	83	28.3	128	53.3	173	78.3
-7	-21.7	39	3.9	84	28.9	129	53.9	174	78.9
-6	-21.1	39.2	4	84.2	29	129.2	54	174.2	79
-5.8	-21								

FAHRENHEIT AND CENTIGRADE SCALES

Messrs Negretti & Zambra)

°F	°C	°F	°C	°F	°C	°F	°C	°F	°C
175	79.4	220	104.4	265	129.4	310	154.4	355	179.4
176	80	221	105	266	130	311	155	356	180
177	80.6	222	105.6	267	130.6	312	155.6	357	180.6
177.8	81	222.8	106	267.8	131	312.8	156	357.8	181
178	81.1	223	106.1	268	131.1	313	156.1	358	181.1
179	81.7	224	106.7	269	131.7	314	156.7	359	181.7
179.6	82	224.6	107	269.6	132	314.6	157	359.6	182
180	82.2	225	107.2	270	132.2	315	157.2	360	182.2
181	82.8	226	107.8	271	132.8	316	157.8	361	182.8
181.4	83	226.4	108	271.4	133	316.4	158	361.4	183
182	83.3	227	108.3	272	133.3	317	158.3	362	183.3
183	83.9	228	108.9	273	133.9	318	158.9	363	183.9
183.2	84	228.2	109	273.2	134	318.2	159	363.2	184
184	84.4	229	109.4	274	134.4	319	159.4	364	184.4
185	85	230	110	275	135	320	160	365	185
186	85.6	231	110.6	276	135.6	321	160.6	366	185.6
186.8	86	231.8	111	276.8	136	321.8	161	366.8	186
187	86.1	232	111.1	277	136.1	322	161.1	367	186.1
188	86.7	233	111.7	278	136.7	323	161.7	368	186.7
188.6	87	233.6	112	278.6	137	323.6	162	368.6	187
189	87.2	234	112.2	279	137.2	324	162.2	369	187.2
190	87.8	235	112.8	280	137.8	325	162.8	370	187.8
190.4	88	235.4	113	280.4	138	325.4	163	370.4	188
191	88.3	236	113.3	281	138.3	326	163.3	371	188.3
192	88.9	237	113.9	282	138.9	327	163.9	372	188.9
192.2	89	237.2	114	282.2	139	327.2	164	372.2	189
193	89.4	238	114.4	283	139.4	328	164.4	373	189.4
194	90	239	115	284	140	329	165	374	190
195	90.6	240	115.6	285	140.6	330	165.6	375	190.6
195.8	91	240.8	116	285.8	141	330.8	166	375.8	191
196	91.1	241	116.1	286	141.1	331	166.1	376	191.1
197	91.7	242	116.7	287	141.7	332	166.7	377	191.7
197.6	92	242.6	117	287.6	142	332.6	167	377.6	192
198	92.2	243	117.2	288	142.2	333	167.2	378	192.2
199	92.8	244	117.8	289	142.8	334	167.8	379	192.8
199.4	93	244.4	118	289.4	143	334.4	168	379.4	193
200	93.3	245	118.3	290	143.3	335	168.3	380	193.3
201	93.9	246	118.9	291	143.9	336	168.9	381	193.9
201.2	94	246.2	119	291.2	144	336.2	169	381.2	194
202	94.4	247	119.4	292	144.4	337	169.4	382	194.4
203	95	248	120	293	145	338	170	383	195
204	95.6	249	120.6	294	145.6	339	170.6	384	195.6
204.8	96	249.8	121	294.8	146	339.8	171	384.8	196
205	96.1	250	121.1	295	146.1	340	171.1	385	196.1
206	96.7	251	121.7	296	146.7	341	171.7	386	196.7
206.6	97	251.6	122	296.6	147	341.6	172	386.6	197
207	97.2	252	122.2	297	147.2	342	172.2	387	197.2
208	97.8	253	122.8	298	147.8	343	172.8	388	197.8
208.4	98	253.4	123	298.4	148	343.4	173	388.4	198
209	98.3	254	123.3	299	148.3	344	173.3	389	198.3
210	98.9	255	123.9	300	148.9	345	173.9	390	198.9
210.2	99	255.2	124	300.2	149	345.2	174	390.2	199
211	99.4	256	124.4	301	149.4	346	174.4	391	199.4
212	100	257	125	302	150	347	175	392	200
213	100.6	258	125.6	303	150.6	348	175.6	393	200.6
213.8	101	258.8	126	303.8	151	348.8	176	393.8	201
214	101.1	259	126.1	304	151.1	349	176.1	394	201.1
215	101.7	260	126.7	305	151.7	350	176.7	395	201.7
215.6	102	260.6	127	305.6	152	350.6	177	395.6	202
216	102.2	261	127.2	306	152.2	351	177.2	396	202.2
217	102.8	262	127.8	307	152.8	352	177.8	397	202.8
217.4	103	262.4	128	307.4	153	352.4	178	397.4	203
218	103.3	263	128.3	308	153.3	353	178.3	398	203.3
219	103.9	264	128.9	309	153.9	354	178.9	399	203.9
219.2	104	264.2	129	309.2	154	354.2	179	399.2	204

TABLE 50: CONVERSION TABLE FOR
(With acknowledgments to

°F	°C	°F	°C	°F	°C	°F	°C	°F	°C
400	204.4	445	229.4	490	254.4	535	279.4	580	304.4
401	205	446	230	491	255	536	280	581	305
402	205.6	447	230.6	492	255.6	537	280.6	582	305.6
402.8	206	447.8	231	492.8	256	537.8	281	582.8	306
403	206.1	448	231.1	493	256.1	538	281.1	583	306.1
404	206.7	449	231.7	494	256.7	539	281.7	584	306.7
404.6	207	449.6	232	494.6	257	539.6	282	584.6	307
405	207.2	450	232.2	495	257.2	540	282.2	585	307.2
406	207.8	451	232.8	496	257.8	541	282.8	586	307.8
406.4	208	451.4	233	496.4	258	541.4	283	586.4	308
407	208.3	452	233.3	497	258.3	542	283.3	587	308.3
408	208.9	453	233.9	498	258.9	543	283.9	588	308.9
408.2	209	453.2	234	498.2	259	543.2	284	588.2	309
409	209.4	454	234.4	499	259.4	544	284.4	589	309.4
410	210	455	235	500	260	545	285	590	310
411	210.6	456	235.6	501	260.6	546	285.6	591	310.6
411.8	211	456.8	236	501.8	261	546.8	286	591.8	311
412	211.1	457	236.1	502	261.1	547	286.1	592	311.1
413	211.7	458	236.7	503	261.7	548	286.7	593	311.7
413.6	212	458.6	237	503.6	262	548.6	287	593.6	312
414	212.2	459	237.2	504	262.2	549	287.2	594	312.2
415	212.8	460	237.8	505	262.8	550	287.8	595	312.8
415.4	213	460.4	238	505.4	263	550.4	288	595.4	313
416	213.3	461	238.3	506	263.3	551	288.3	596	313.3
417	213.9	462	238.9	507	263.9	552	288.9	597	313.9
417.2	214	462.2	239	507.2	264	552.2	289	597.2	314
418	214.4	463	239.4	508	264.4	553	289.4	598	314.4
419	215	464	240	509	265	554	290	599	315
420	215.6	465	240.6	510	265.6	555	290.6	600	315.6
420.8	216	465.8	241	510.8	266	555.8	291	600.8	316
421	216.1	466	241.1	511	266.1	556	291.1	601	316.1
422	216.7	467	241.7	512	266.7	557	291.7	602	316.7
422.6	217	467.6	242	512.6	267	557.6	292	602.6	317.2
423	217.2	468	242.2	513	267.2	558	292.2	603	317.2
424	217.8	469	242.8	514	267.8	559	292.8	604	317.8
424.4	218	469.4	243	514.4	268	559.4	293	604.4	318
425	218.3	470	243.3	515	268.3	560	293.3	605	318.3
426	218.9	471	243.9	516	268.9	561	293.9	606	318.9
426.2	219	471.2	244	516.2	269	561.2	294	606.2	319
427	219.4	472	244.4	517	269.4	562	294.4	607	319.4
428	220	473	245	518	270	563	295	608	320
429	220.6	474	245.6	519	270.6	564	295.6	609	320.6
429.8	221	474.8	246	519.8	271	564.8	296	609.8	321
430	221.1	475	246.1	520	271.1	565	296.1	610	321.1
431	221.7	476	246.7	521	271.7	566	296.7	611	321.7
431.6	222	476.6	247	521.6	272	566.6	297	611.6	322
432	222.2	477	247.2	522	272.2	567	297.2	612	322.2
433	222.8	478	247.8	523	272.8	568	297.8	613	322.8
433.4	223	478.4	248	523.4	273	568.4	298	613.4	323
434	223.3	479	248.3	524	273.3	569	298.3	614	323.3
435	223.9	480	248.9	525	273.9	570	298.9	615	323.9
435.2	224	480.2	249	525.2	274	570.2	299	615.2	324
436	224.4	481	249.4	526	274.4	571	299.4	616	324.4
437	225	482	250	527	275	572	300	617	325
438	225.6	483	250.6	528	275.6	573	300.6	618	325.6
438.8	226	483.8	251	528.8	276	573.8	301	618.8	326
439	226.1	484	251.1	529	276.1	574	301.1	619	326.1
440	226.7	485	251.7	530	276.7	575	301.7	620	326.7
440.6	227	485.6	252	530.6	277	575.6	302	620.6	327
441	227.2	486	252.2	531	277.2	576	302.2	621	327.2
442	227.8	487	252.8	532	277.8	577	302.8	622	327.8
442.4	228	487.4	253	532.4	278	577.4	303	622.4	328
443	228.3	488	253.3	533	278.3	578	303.3	623	328.3
444	228.9	489	253.9	534	278.9	579	303.9	624	328.9
444.2	229	489.2	254	534.2	279	579.2	304	624.2	329

PHYSICAL DATA

403

FAHRENHEIT AND CENTIGRADE SCALES—continued

Messrs Negretti & Zambra)

°F	°C	°F	°C	°F	°C	°F	°C	°F	°C
625	329.4	670	354.4	715	379.4	760	404.4	805	429.4
626	330	671	355	716	380	761	405	806	430
627	330.6	672	355.6	717	380.6	762	405.6	807	430.6
627.8	331	672.8	356	717.8	381	762.8	406	807.8	431
628	331.1	673	356.1	718	381.1	763	406.1	808	431.1
628.6	331.7	673.6	356.7	718.6	381.7	764	406.7	809	431.7
629	332	674	357	719	382	764.6	407	809.6	432
630	332.2	675	357.2	720	382.2	765	407.2	810	432.2
631	332.8	676	357.8	721	382.8	766	407.8	811	432.8
631.4	333	676.4	358	721.4	383	766.4	408	811.4	433
632	333.3	677	358.3	722	383.3	767	408.3	812	433.3
633	333.9	678	358.9	723	383.9	768	408.9	813	433.9
633.2	334	678.2	359	723.2	384	768.2	409	813.2	434
634	334.4	679	359.4	724	384.4	769	409.4	814	434.4
635	335	680	360	725	385	770	410	815	435
636	335.6	681	360.6	726	385.6	771	410.6	816	435.6
636.8	336	681.8	361	726.8	386	771.8	411	816.8	436
637	336.1	682	361.1	727	386.1	772	411.1	817	436.1
638	336.7	683	361.7	728	386.7	773	411.7	818	436.7
638.6	337	683.6	362	728.6	387	773.6	412	818.6	437
639	337.2	684	362.2	729	387.2	774	412.2	819	437.2
640	337.8	685	362.8	730	387.8	775	412.8	820	437.8
640.4	338	685.4	363	730.4	388	775.4	413	820.4	438
641	338.3	686	363.3	731	388.3	776	413.3	821	438.3
642	338.9	687	363.9	732	388.9	777	413.9	822	438.9
642.2	339	687.2	364	732.2	389	777.2	414	822.2	439
643	339.4	688	364.4	733	389.4	778	414.4	823	439.4
644	340	689	365	734	390	779	415	824	440
645	340.6	690	365.6	735	390.6	780	415.6	825	440.6
645.8	341	690.8	366	735.8	391	780.8	416	825.8	441
646	341.1	691	366.1	736	391.1	781	416.1	826	441.1
647	341.7	692	366.7	737	391.7	782	416.7	827	441.7
647.6	342	692.6	367	737.6	392	782.6	417	827.6	442
648	342.2	693	367.2	738	392.2	783	417.2	828	442.2
649	342.8	694	367.8	739	392.8	784	417.8	829	442.8
649.4	343	694.4	368	739.4	393	784.4	418	829.4	443
650	343.3	695	368.3	740	393.3	785	418.3	830	443.3
651	343.9	696	368.9	741	393.9	786	418.9	831	443.9
651.2	344	696.2	369	741.2	394	786.2	419	831.2	444
652	344.4	697	369.4	742	394.4	787	419.4	832	444.4
653	345	698	370	743	395	788	420	833	445
654	345.6	699	370.6	744	395.6	789	420.6	834	445.6
654.8	346	699.8	371	744.8	396	789.8	421	834.8	446
655	346.1	700	371.1	745	396.1	790	421.1	835	446.1
656	346.7	701	371.7	746	396.7	791	421.7	836	446.7
656.6	347	701.6	372	746.6	397	791.6	422	836.6	447
657	347.2	702	372.2	747	397.2	792	422.2	837	447.2
658	347.8	703	372.8	748	397.8	793	422.8	838	447.8
658.4	348	703.4	373	748.4	398	793.4	423	838.4	448
659	348.3	704	373.3	749	398.3	794	423.3	839	448.3
660	348.9	705	373.9	750	398.9	795	423.9	840	448.9
660.2	349	705.2	374	750.2	399	795.2	424	840.2	449
661	349.4	706	374.4	751	399.4	796	424.4	841	449.4
662	350	707	375	752	400	797	425	842	450
663	350.6	708	375.6	753	400.6	798	425.6	843	450.6
663.8	351	708.8	376	753.8	401	798.8	426	843.8	451
664	351.1	709	376.1	754	401.1	799	426.1	844	451.1
665	351.7	710	376.7	755	401.7	800	426.7	845	451.7
665.6	352	710.6	377	755.6	402	800.6	427	845.6	452
666	352.2	711	377.2	756	402.2	801	427.2	846	452.2
667	352.8	712	377.8	757	402.8	802	427.8	847	452.8
667.4	353	712.4	378	757.4	403	802.4	428	847.4	453
668	353.3	713	378.3	758	403.3	803	428.3	848	453.3
669	353.9	714	378.9	759	403.9	804	428.9	849	453.9
669.2	354	714.2	379	759.2	404	804.2	429	849.2	454

